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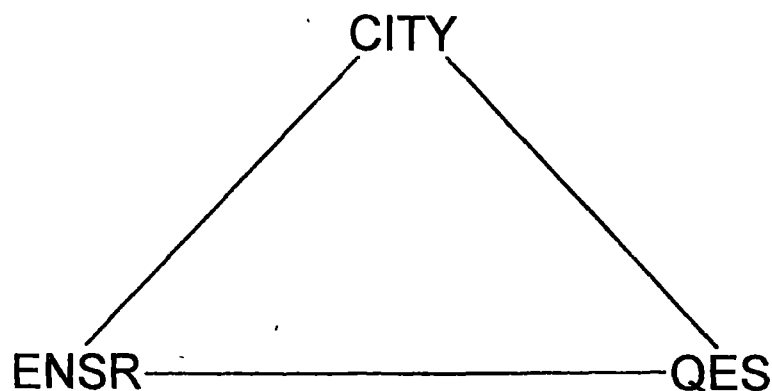
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1998 SAMPLING PLAN
REILLY TAR & CHEMICAL CORP.
N. P. L. SITE
ST. LOUIS PARK, MINNESOTA





October 30, 1997

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

**ENSR Consulting
and Engineering**

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Regional Administrator
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520 Lafayette Road North
St. Paul, Minnesota 55155

President
Reilly Industries, Inc.
300 N. Meridian St., Suite 1500
Indianapolis, Indiana 46204-1763

**Re: United States of America, et al. vs. Reilly Tar & Chemical Corporation, et
al.
File No. Civ. 4-80-469
CD-RAP Section 3.3**


Gentlemen:

In accordance with Section 3.3 of the Remedial Action Plan for the referenced case, the City of St. Louis Park hereby submits the 1998 Sampling Plan.

Additionally, the original signature page to the Quality Assurance Project Plan has been included in the Minnesota Pollution Control Agency's copy of the 1998 Sampling Plan.

Any comments regarding this submittal may be directed to this office.

Sincerely,


William M. Gregg
Project Leader for the
City of St. Louis Park

cc: Mike Rardin
Scott Anderson

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**REILLY TAR AND CHEMICAL CORPORATION
N.P.L. SITE
ST. LOUIS PARK, MINNESOTA
SITE MANAGEMENT PLAN**

INTRODUCTION

Ground water in the City of St. Louis Park, Minnesota, has been found to contain polynuclear aromatic hydrocarbons (PAH) and phenolics as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAHs in various aquifers beneath St. Louis Park and adjacent communities.

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Agency (MPCA), the Minnesota Department of Health (MDH), the City of St. Louis Park (City), and Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into a Consent Decree, include the following concentration levels:

	Advisory Level	Drinking Water Criteria
Sum of benzo(a)pyrene and dibenz(a,h)anthracene	3.0 ng/l*	5.6 ng/l
Carcinogenic PAH	15 ng/l	28 ng/l
Other PAH	175 ng/l	280 ng/l

* or the lowest concentration that can be quantified, whichever is greater

In conjunction with the implementation of remedial measures to limit the spread of PAH and phenolics, granular activated carbon (GAC) treatment systems have been installed to treat water from City wells (identified - SLP) 4, 10 and 15. Further provisions of a Remedial Action Plan (RAP) call for long-term monitoring of the influent and effluent of the GAC treatment systems and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water. The analytical data will be used to evaluate water quality by comparing the levels of PAH and/or phenolics found in the various samples with historical water quality data and with water quality criteria established in the Consent Decree-RAP. The specific objectives of the monitoring program, and therefore, the intended end use of the data vary slightly for the different aquifers being monitored in accordance with the Consent Decree-RAP.

The objective of the GAC treatment system monitoring is to assess and evaluate the performance of the treatment systems. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the Consent Decree-RAP. Based on these comparisons, decisions will be made on: 1) system operations (e.g., when the carbon should be replaced), and 2) cessation of the treatment systems, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of monitoring the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH, is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the Site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the Consent Decree-RAP.

If any new Iron-ton-Galesville Aquifer drinking water wells are installed within one mile of well W23, then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH found in potential Iron-ton-Galesville Aquifer drinking water wells to the drinking water criteria established in the Consent Decree-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking wells, private or industrial wells, and monitoring wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the Consent Decree-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23). Water level data will be used to evaluate ground water flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to: 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the Consent Decree-RAP. Water level data will be used to evaluate ground water patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells is to monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems. Ground water analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the Consent Decree-RAP for PAH and phenolics. Water level data will be used to evaluate ground water flow patterns in the Drift-Platteville Aquifer.

The Site Management Plan (Plan) outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, Minnesota, area in accordance with the Consent Decree-RAP related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for ground water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. Although a GAC treatment system has been constructed to treat water from wells W23, W105, and the Drift-Platteville Aquifer source control wells prior to its discharge to surface water receivers, monitoring of the effluent is not within the scope of work to be

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performed under this Plan, as the activity is not embodied in the Consent Decree-RAP. Similarly, a GAÇ treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the Consent Decree-RAP.

The time period covered by this Plan is from January 1, 1998, or the date of its acceptance and approval by the Agencies whichever is later, to December 31, 1998. The next subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1998 covering the 1999 calendar year.

This Plan incorporates the requirements of RAP Sections 3.2, 3.3, 4.3, 5.1, 7.3, 8.1.3, 9.1.3, 9.2.3, 9.3.3, and 9.6. Some of the monitoring required under these RAP Sections has already taken place in accordance with previous Sampling Plans.

MONITORING SCHEDULE

The monitoring schedule outlined in this Plan indicates the starting criteria and the frequencies of monitoring as outlined in the RAP to determine when the GAC treatment system and wells are monitored (Tables 1 and 2). In general, the monitoring schedule will allow economies of scale in the field and in the laboratory by grouping the various monitoring events described by the RAP as much as possible. Samples will be collected within the time periods indicated on Tables 1 and 2, and all parties will be given at least 48 hours notice in advance of routine sampling.

Tables 1 and 2 summarize the GAC system/ground water monitoring schedule for the period through December 1998, and represent the minimum monitoring program that is likely to occur during the year. However, additional monitoring will take place if treated water from the GAC treatment system or ground water from active municipal drinking water wells exceeds the drinking water criteria established in the Consent Decree-RAP. This additional monitoring is described in Sections 4 and 12 of the RAP, and are reproduced in Appendix A of this Plan.

The duration of field sampling events will depend on the number and type of wells to be monitored. For estimating purposes, Drift and Platteville Aquifer monitoring wells typically are monitored at a rate of five to 10 wells per day, St. Peter Aquifer monitoring wells typically are monitored at a rate of five wells per day, and Prairie du Chien Aquifer monitoring wells typically require two to four hours or more per well to monitor.

TABLE 1

Sampling Plan GAC Treatment System Monitoring Schedule^a

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses^b
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH(ppt) ^c
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625
<p>a This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1998. Sections 4 and 12 of the RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC treatment system is shown in Figure 1.</p> <p>b Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one every ten samples or fewer. Treated water will be duplicated at a rate of 100 percent. Feed water duplicate samples will be collected and analyzed at a frequency of one per ten samples.</p> <p>c ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.</p>				

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TABLE 2
Sampling Plan Ground Water Monitoring Schedule^a

Source of Water	RAP Section	Sampling ^b Points	Start of Monitoring	Sampling Frequency	Analyses ^c
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP 17	Date of plan approval	Annually	PAH(ppt) ^d
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Ironton-Galesville Aquifer	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Prairie du Chien-Jordan Aquifer	7.3(A)	SLP4	Start of pumping	Semi-annually	PAH(ppt) phenolics
	7.3(B)	W23	Date of plan approval	Semi-annually	PAH(ppb) ^e
	7.3(C)	SLP6, SLP7 or SLP9	Date of plan approval	Annually	PAH(ppt)
	7.3(D)	W405 or W406 ^f , H3, SLP10 or SLP15, SLP14, SLP16, W402 W403, W119	Date of plan approval	Annually	PAH(ppt)
	7.3(E)	SLP5, H6, E3, MTK6, W29, W40, W70	Date of plan approval	Annually	PAH(ppt)
	7.3(F) ^g	W32, SLP8, SLP10, E4	Date of plan approval	Semi-annually	No chemical analyses ^g
	7.4.1 ^h	W48, W401, E2, E7, E13, E15	Date of plan approval	Semi-annually	PAH(ppt)
St. Peter Aquifer	8.1.3 ⁱ	SLP3, W24, W33, W122, W129, W133, W408, W409, W410, W411, W412, P116	Date of plan approval	Semi-annually	PAH(ppt)

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TABLE 2

Sampling Plan Ground Water Monitoring Schedule^a

Source of Water	RAP Section	Sampling^b Points	Start of Monitoring	Sampling Frequency	Analyses^c
Drift-Platteville Aquifer	9.1.3 and 9.2.3	W420, W421, W422, W439	Date of plan approval	Quarterly	PAH(ppb) and total phenols
	9.5	W1, W18, W19, W20, W22, W27, W101, W120, W121, W124, W130, W131, W143, W424, W426, W428, W431, W432, W433, W434, W440	Date of plan approval	Semi-annually	PAH(ppt)

TABLE 2

Sampling Plan Ground Water Monitoring Schedule^a

Source of Water	RAP Section	Sampling ^b Points	Start of Monitoring	Sampling Frequency	Analyses ^c
a	This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1998. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one for every ten samples or fewer, and one duplicate sample will be collected for every ten samples.				
b	Sampling points are located on the maps shown in Figures 1 through 5. Letter prefixes to well codes are defined as follows:				
	W	4-inch monitoring well			
	P	monitoring piezometer			
	SLP	St. Louis Park supply well			
	E	Edina supply well			
	H	Hopkins supply well			
	MTK	Minnetonka supply well			
c	Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analyses, except for those wells which prove to be inaccessible for such measurements.				
d	ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.				
e	ppb = parts per billion. This signifies analysis by the Non-Criteria Method. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the Low-Level Method will be used on subsequent monitoring rounds.				
f	W405 = American Hardware Mutual, W406 = Minikahda Golf Course.				
g	Water levels will be measured semi-annually at these wells, except for those wells which prove to be inaccessible for such measurements.				
h	In accordance with the Gradient Control Modification System, these wells are now sampled semi-annually as opposed to annually.				
i	Section 8.1.3 of the Consent Decree-RAP originally specified St. Peter Aquifer monitoring requirements. Monitoring requirements for 1998, and subsequent years are now specified in the St. Peter Aquifer Record of Decision (ROD).				
j	These wells were requested to be sampled semi-annually in accordance with the ROD for the Northern Area of the Platteville Aquifer. However, three of the wells, W420, W421, and W422, are required to be sampled quarterly per Section 9.1.3 and 9.2.3 and will continue to be sampled quarterly. Additionally, SLP3 is already required to be sampled semi-annually per Section 8.1.3 and will continue to be sampled twice per year.				

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FIGURE 4 LOCATIONS OF DRIFT/PLATTEVILLE AQUIFER WELLS

GROUND WATER SAMPLING PROCEDURES

An important distinction is made between the sampling procedures for active pumping wells (e.g. municipal wells) and for non-pumping monitoring wells. Active pumping wells are used on a regular basis, have dedicated pumps and associated plumbing, and have sample taps for collecting samples. Non-pumping monitoring wells may be new, or may have not been pumped for several years, and most require pumping and associated equipment for sampling. Another distinction is that the active pumping wells are typically located inside buildings whereas non-pumping monitoring wells are not.

With these considerations in mind, this Plan has been developed so that the ground water monitoring program in each aquifer meets the requirements and intent of the RAP. Ground water monitoring will be conducted in accordance with the procedures given in the Quality Assurance Project Plan (QAPP), and with Minnesota Pollution Control Agency guidelines entitled "Development of Sampling Plans, Protocols and Reports", January 1995.

Water Level Measurements

Water level measurements will be made using electric tapes or weighted steel tapes. Water level measurements using steel tapes will be made by suspending a known length of tape in the well so that the bottom end of the tape is below the water level. The lower portion of tape will be coated with blue chalk that exhibits a noticeable color change when wetted. The water level measurement will be obtained by subtracting the length of wetted tape from the total length of tape suspended below the measuring point of each well.

Using the electric tape, the probe at the end of the tape will be lowered slowly in the well until contact with the water is made. Because of surface tension, readings of the water level made when the probe enters the water will differ from readings made when the probe leaves the water, thus breaking surface tension. To standardize these measurements, the second reading will always be used (i.e. the reading made when the probe leaves the water).

Water level measurement made for the purpose of defining ground water flow patterns in a particular aquifer may be performed independently from ground water sampling, as a discrete event so as not to last more than two days. The wells will be revisited for sampling, and measurements to determine the volume of water in the well will be made at that time.

Sample Collection at Active Pumping Wells

At active pumping wells, the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. An electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered. The only exception to this is the GAC treatment system monitoring under RAP Section 4.3 which includes treated water monitoring.

If it cannot be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sample Collection at Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in the purging and sampling of the monitoring wells and piezometers, flexibility in the approach to sample retrieval is necessary. This Plan proposes that the sampling team be given latitude in the selection of purge/sample equipment and procedures necessary to complete the monitoring task.

Table 2 specifies the monitoring of Prairie du Chien-Jordan Aquifer monitor well W70 which is equipped with an operable dedicated submersible pump. Well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with parameter monitoring established herein.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a non-dedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH, and specific conductance of the purge water will be monitored using a Horiba water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without pre-rinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

The discharge from purging monitoring wells will be handled in accordance with the Contingency Plan (Appendix B). In general, if a visible sheen can be seen on the water surface, the discharge will be routed to the sanitary sewer. Otherwise, the storm sewer or surface water discharge will be used. Non-dedicated ground water sampling or monitoring equipment that comes in contact with the ground water will be decontaminated between uses, as described in the QAPP.

ANALYTICAL PROGRAM

Tables 1 and 2 show the ground water monitoring summary as prescribed in the RAP. Indicated on the tables are the analyses required. Details of all analytical methodology can be found in the QAPP and its appendices. All analyses will be performed at Quanterra Incorporated's Arvada, Colorado, analytic facility. Quanterra has agreed to provide a turnaround time of 30 working days from the receipt of samples to the submittal of analytical reports. The laboratory will notify the City if it cannot meet this turnaround time.

Ground water monitoring will include two methods of PAH analyses depending upon the anticipated PAH concentration levels. Low-Level (nanograms per liter or part per trillion) PAH analyses will be performed utilizing selected ion monitoring (SIM) gas chromatography mass spectrometry (GC/MS). This method will be used to analyze samples from drinking water wells and from other wells for which the RAP requires drinking water criteria to be enforced (e.g. St. Peter Aquifer monitoring wells). This method is designed to analyze samples containing up to 600 nanograms per liter of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the microgram per liter range. Specific details of this methodology can be found in Appendix B of the QAPP.

Non-criteria level (micrograms per liter or part per billion) PAH analyses, using the Scanning GC/MS Method, will be performed on samples from wells that have historically contained elevated PAH concentrations (e.g. part per million levels in well W23), and on wells that are not subject to the RAP's requirements for meeting drinking water criteria (e.g. Drift-Platteville Aquifer monitoring wells).

Two methods are required for PAH analyses because the Low-Level part per trillion SIM method is not appropriate for samples containing more than approximately 20 micrograms per liter of total PAH. Analysis of samples containing total PAH concentrations over 20 micrograms per liter, if performed with the Low-Level method, requires multiple dilutions and increases the risk of cross-contamination of the samples. This decreases the reliability of the data. Not only will multiple dilutions increase the variability of measurements, but critical quality control information (e.g., surrogate recoveries) is lost. Therefore, for samples containing greater than 20 micrograms per liter of total PAH, the analytical method that will be used is Scanning GC/MS Method as described in the QAPP.

The Scanning GC/MS Method analysis will be performed on 1-liter samples, and will have detection limits of 10 micrograms per liter. For wells that are tested with this Non-Criteria method, if the analytical results of historical monitoring indicate total PAH concentrations less than 20 micrograms per liter, the Low-Level method will be used to analyze samples in 1998. This procedure will allow an evaluation of long-term PAH concentrations around the fringe PAH contamination in the Drift-Platteville Aquifer.

Depending on the circumstances and the actual PAH level, previous analytical results using the Low-Level that exceed 20,000 nanograms per liter of total PAH will indicate a switch to the Scanning GC/MS Method for 1998 sampling rounds.

REPORTING

The analytical reporting requirements of the Consent Decree and RAP are identified in Part K of the Consent Decree, and Sections 3.4, 4.3.5, 12.1.1, and 12.1.2 of the RAP. Part K requires Reilly to submit an annual progress report on March 15, 1998. This report will contain analytical reports as specified in Section 5.0 of the QAPP for this Plan, all water level measurements and chemical analyses that have not been presented in previous reports, and interpretive maps and tables, as specified in RAP Section 3.4(B) and (C). Also, the effectiveness of the source and gradient control well systems in the Drift-Platteville and St. Peter Aquifers will be discussed in the annual report.

The reporting requirement for each aquifer, and for the GAC treatment system, are described below.

GAC Treatment System

RAP Section 4.3.5 requires the City to submit an annual report that presents the results of all monitoring of the GAC treatment system. Analytical results for wellhead water, feed water, and treated water will be included in this report. The report will also describe briefly the operating performance of the GAC treatment system during the previous calendar year. The GAC treatment system annual reports are due each March 15.

Mt. Simon-Hinckley Aquifer

The monitoring data for the Mt. Simon-Hinckley Aquifer will be included in the annual report. In addition to the results of all water level measurements and chemical analyses, the report will contain a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well in accordance with RAP Section 3.4(C). Since the Mt. Simon-Hinckley Aquifer wells are monitored on an annual basis, there will be only one sampling event to report.

Ironton-Galesville Aquifer

The monitoring data for the Ironton-Galesville Aquifer will be included in the Annual Report, if any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23.

Prairie du Chien-Jordan Aquifer

The monitoring data for the Prairie du Chien-Jordan Aquifer will be included in the annual report. The results of all water level measurements and chemical analyses will be included. For each of the water level measuring periods, a water level contour map will be prepared with elevations labelled at each well. For each sampling event, a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well will be prepared in accordance with

RAP Section 3.4(C), and a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

St. Peter Aquifer

The monitoring data for the St. Peter Aquifer will be included in the annual report. The results of chemical analyses will be reported and a map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well will be prepared in accordance with RAP Section 3.4(C). Likewise, the results of water level measurements will be provided and a water level contour map will be prepared with elevations labelled at each well in accordance with RAP Section 3.4(B). In addition, a map of the area indicating the extent of PAH above drinking water criteria shall be provided.

Drift-Platteville Aquifer

The monitoring data for the Drift-Platteville Aquifer including the results of all water level measurements and chemical analyses, will be presented in the Annual Monitoring Report. A map showing each well sampled with the concentrations of Other PAH, Carcinogenic PAH, and the sum of benzo(a)pyrene and dibenz(a,h) anthracene labelled by the location of each well, and a map with phenolics concentrations labelled by the location of each well will be prepared in accordance with RAP Section 3.4. The Drift-Platteville Aquifer monitoring data will be included in the annual report to support a discussion of the results with respect to the effectiveness of the source and gradient control well systems.

A



APPENDIX A
ADDITIONAL MONITORING REQUIREMENTS

Level or Drinking Water Criterion is exceeded during the first year of operation of the system, Reilly shall immediately notify the Regional Administrator, the Director, and the Commissioner, and shall undertake such additional Monitoring as is required by Section 4.3.2.

- (D) Routine Monitoring after two carbon changes shall be quarterly, unless the Regional Administrator, the Director, and the Commissioner determine that the observed service life of the carbon is too short to permit this frequency, in which case the Regional Administrator, the Director and the Commissioner shall notify Reilly of the required Monitoring frequency in accordance with Part G or H of the Consent Decree.

4.3.2. Carbon Replacement Monitoring

- (A) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Drinking Water Criterion for Other PAH or exceed the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall collect two additional treated water samples at least 2 Days apart within one week of receiving the results of the exceedance sample. If the

analytical results from either one or both of the two additional samples also exceed the Drinking Water Criterion for Other PAH or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(B) If the analytical results from any treated water sample obtained pursuant to Section 4.3.1. exceed the Advisory Level for Other PAH, then Monitoring of treated water shall be conducted immediately according to Section 12.1. If the results of any two samples required by Section 12.1. exceed the Drinking Water Criterion for Other PAH, and neither of the conditions specified in (C)(1) and (2) below are met, then the carbon shall be replaced within 21 Days of receiving the additional sample results.

(C) If any analytical result from the additional samples taken as required by (A) or (B) above exceeds the Drinking Water Criterion for Other PAH, or the Advisory Level for either Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene during either

- (1) within one year after the carbon treatment system is placed into service or
- (2) within one year after the first carbon change if carbon was changed in the first year of operation of the carbon treatment system,

then Reilly shall conduct the Monitoring program specified in Section 4.6. Reilly shall report the results of the Section 4.6. Monitoring program to the Regional Administrator, the Director and the Commissioner within 7 Days of receiving the analytical data. If the treated water from the carbon treatment system is determined pursuant to Section 4.6. to exceed the Drinking Water Criterion for Other PAH or the Advisory Levels for Carcinogenic PAH or the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then Reilly shall replace the carbon within 14 Days of making this determination. If the treated water is determined pursuant to Section 4.6. to meet the Drinking Water Criterion for Other PAH and the Advisory Levels for Carcinogenic PAH and the sum of benzo(a)pyrene and dibenz(a,h)anthracene, then normal GAC system operation and Monitoring in accordance with Sections 4.3.1.(B) and

- (C) After the first month of operation, Monitoring of feed water shall be performed quarterly until the carbon has been changed twice. If the Regional Administrator, the Director and the Commissioner determine pursuant to Section 4.3.1.(2) that the GAC system is not operating properly, Reilly may, upon receipt of such determination, be required to resume biweekly Monitoring of feed water.
- (D) After two carbon changes in the GAC system, feed water shall be Monitored annually.

4.3.4. Extended Monitoring

Treated water from the GAC system shall be sampled and analyzed annually for the extended list of PAH in Part A.2. of Appendix A, using gas chromatography/mass spectroscopy (GC/MS), or other methods approved by the Regional Administrator and the Director. During this extended analysis, any compounds listed in Part A.2. of Appendix A, or any other compounds which are detected with significant peak heights that are not routinely Monitored, shall be identified and, if possible, quantified, using a mass spectral library which contains extensive spectra of PAH compounds, such as the National Bureau of Standards mass spectral library. Reilly shall analyze a sample of treated or feed water once a year for the acid fraction compounds determined by EPA Test Method 625 or by other methods approved by the Regional Administrator and the Director.

CONTINGENT ACTIONS FOR MUNICIPAL
DRINKING WATER SUPPLY WELLS

12.1. Contingent Monitoring

12.1.1. Exceedance of Advisory Levels

If the analytical result of any sample taken from an active municipal drinking water well under the Monitoring requirements of Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above exceeds an Advisory Level, Reilly shall take another sample within seven Days of receiving the analytical results and analyze this sample. If the results of the second sample are below all of the Advisory Levels, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample. If the third sample is below all of the Advisory Levels, Monitoring of the affected well shall revert to its normal schedule. If the analytical result of the second or third sample exceeds an Advisory Level but is less than all Drinking Water Criteria, the Regional Administrator, the Director, and the Commissioner shall be notified by Reilly immediately and subsequent samples shall be taken by Reilly monthly until such time as either:

- (A) three consecutive samples yield results less than all of the Advisory Levels, in which case the sampling interval shall revert to the level specified for the affected well in Sections 3., 4.3., 5.1., 6.2.1., 7.3., or 8.4. above; or

- (B) a sample yields results greater than a Drinking Water Criterion, in which case the requirements of Section 12.1.2., below, apply.

12.1.2. Exceedance of Drinking Water Criteria

- (A) If the analytical result of any sample taken from an active municipal drinking water well pursuant to Section 12.1.1 exceeds the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, the Regional Administrator, the Director and the Commissioner shall be immediately notified by Reilly, and another sample shall be taken by Reilly within three Days of receiving the results of the first sample and analyzed. If the analytical result of the second sample is less than all of the Drinking Water Criteria but greater than any Advisory Level, a third sample shall be taken by Reilly within seven Days of receiving the results of the second sample and analyzed. If the results of this third sample are less than all of the Drinking Water Criteria, but greater than any Advisory Level, Reilly shall comply with the monthly sampling frequency specified in Section 12.1.1. above.

(B) If the analytical result of the second or third sample taken pursuant to Section 12.1.2.(A) above is greater than the Drinking Water Criterion for Carcinogenic PAH, the sum of benzo(a)pyrene and dibenz(a,h)anthracene, or Other PAH, Reilly shall Monitor the well weekly until such time as either: (1) three consecutive samples yield results below all of the Drinking Water Criteria, in which case Monitoring of the well shall revert to the normal schedule (including Advisory Level Monitoring as specified by Section 12.1.1. above if applicable); or, (2) three consecutive samples yield results above any Drinking Water Criterion, in which case Reilly shall immediately notify the Regional Administrator, the Director and the Commissioner. The Commissioner may then require the affected well to be taken out of service, in which case Reilly shall undertake the contingent actions specified in Section 12.2. below.

12.1.3. Analytical Turn-around Time

All Monitoring conducted pursuant to Section 12.1. shall be on a 21-Day turn-around time basis in accordance with Section 2.8.

B

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APPENDIX B
CONTINGENCY PLAN

Contingent Actions for Contaminated Water

It is possible that groundwater contaminated with coal tar materials will be encountered during the sample retrieval operations. Groundwater generated during sample retrieval operations will be classified as contaminated if the water exhibits a discernible oil sheen or oil phase. Contaminated water will be pumped to the sanitary sewer if it contains less than 10 percent organic material. Estimates of flow rate, disposal volume and water quality will be established and the Metropolitan Council Environmental Services (MCES), formerly Metropolitan Waste Control Commission, will be informed before the discharge to the sanitary sewer if the estimated flow exceeds 150 gallons per workday from any individual site. Contaminated liquids containing more than 10 percent organic material or failing to review MCES approval for discharge will be disposed of in accordance with all applicable local, state and federal rules and regulations and Part T of the Consent Decree. Uncontaminated water will be disposed of in the storm sewer or by other means acceptable to the City of St. Louis Park.

The City will be responsible for keeping the Environmental Protection Agency, Minnesota Pollution Control Agency and Reilly Tar & Chemical Corporation informed of all significant actions involving the generation of contaminated groundwater. All actions, decisions and communications by the City, Environmental Protection Agency, Minnesota Pollution Control Agency, and Reilly in dealing with contaminated soils will be in accordance with and subject to the provisions of Parts I, J, and O of the Consent Decree in the Reilly settlement.

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QUALITY ASSURANCE PROJECT PLAN FOR SAMPLING AND ANALYSIS - GROUND WATER AND GAC TREATMENT SYSTEM MONITORING

for the
Reilly Tar & Chemical Corporation
N.P.L. Site
St. Louis Park, Minnesota

Prepared by

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3.0 PROJECT DESCRIPTION

3.1 Background

Ground water in the City of St. Louis Park (City), Minnesota, has been found to contain polynuclear aromatic hydrocarbons (PAH) and phenolics as a result of activities at a coal-tar distillation and wood preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAHs in various aquifers beneath St. Louis Park and adjacent communities. Accordingly, the site of the plant operations was placed on the National Priorities List and the federal and state governments sought remediation of environmental contamination via United States District Court Case No. Civil 4-80-469. A more detailed explanation of site background is contained on Pages 3 through 9 of the Consent Decree. The City's consulting company is ENSR. ENSR works with the City to address issues concerning the Consent Decree - Remedial Action Plan (CD-RAP) which includes work plan development and implementation for various tasks, ground water sampling, and compliance to the CD-RAP.

A summary of the aquifers which underlie the former wood preserving plant site, their approximate location below the surface level, the general use of the aquifers, and the relative maximum historical PAH and phenolics concentrations measured in each unit (as indicated by historical records and the federal government's Record of Decision in Case No. Civil 4-80-469) are as follows:

Aquifer	Approximate Depth (ft.)	Use	Approximate Upper Concentration of	
			Total PAHs	Phenolics
Drift-Platteville	0 - 90	Private/Industrial/Monitor wells	1000 µg/ℓ off site	10,000 µg/ℓ off site
St. Peter	90 - 200	Municipal/Private drinking water wells	10 ng/ℓ off site	16 µg/ℓ off site
Prairie du Chien-Jordan	250-500	Municipal drinking water wells	10 µg/ℓ off site	10 µg/ℓ off site
Ironton-Galesville	700 - 750	Industrial	1.4 µg/ℓ on site	5 µg/ℓ off site
Mt. Simon-Hinckley	800 - 1100	Municipal drinking water wells	16 ng/ℓ off site	Not detected

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More extensive information relative to the identified level of PAHs in the various aquifers is provided in the following reports:

- Annual Monitoring Reports for 1988 through 1996
- St. Peter Aquifer Remedial Investigation Report (March 30, 1989)
- Drift-Platteville Aquifer (Northern Area) Remedial Investigation Report (March 30, 1989)

The United States Environmental Protection Agency (EPA), the Minnesota Pollution Control Authority (MPCA), the Minnesota Department of Health (MDH), the City, and Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly) have agreed to acceptable water quality criteria for PAH. These criteria, as incorporated into the CD- RAP, in the case referenced above, include the following concentration levels:

	Advisory Level	Drinking Water Criteria
Sum of benzo(a)pyrene and dibenz(a,h)anthracene	3.0 ng/ℓ*	5.6 ng/ℓ
Carcinogenic PAH	15 ng/ℓ	28 ng/ℓ
Other PAH	175 ng/ℓ	280 ng/ℓ

* or the lowest concentration that can be quantified, whichever is greater

Table 3-1 lists the nominal reporting limits for the target compounds listed in the CD-RAP. Currently, only Quanterra Environmental Services (QES) has conducted laboratory analyses of ground water samples.

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from City wells (identified - SLP) 10 and 15. Further provisions of the RAP call for long-term monitoring of the influent and effluent of the GAC treatment system and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH and/or phenolics in the ground water and compare the analytical data with water quality criteria established in the CD-RAP. Currently, both the City and ENSR are collecting the ground water samples. Typically, the City collects water samples from pumping wells (i.e. City owned wells) and ENSR collects water samples from non-pumping wells (i.e. monitoring wells). The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data

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TABLE 3-1

Table of Reporting Limits for Tested Parameters

CAS Number	Compound	Reporting Limit ng/L (PPT)	Reporting Limit ug/L (PPB)
271-89-6	2,3-Benzofuran	5.1	10
496-11-7	2,3-Dihydroindene	5.0	10
95-13-6	1H-Indene	0.9	10
91-20-3	Naphthalene	6.5	10
4565-32-6	Benzo(b)thiophene	0.9	10
91-22-5	Quinoline	6.9	10
120-72-9	1H-Indole	2.5	10
91-57-6	2-Methylnaphthalene	3.9	10
90-12-0	1-Methylnaphthalene	2.8	10
92-52-4	Biphenyl	4.3	10
208-96-8	Acenaphthylene	1.4	10
83-32-9	Acenaphthene	1.3	10
132-64-9	Dibenzofuran	1.0	10
86-73-7	Fluorene	1.0	10
132-65-0	Dibenzothiophene	1.1	10
85-01-8	Phenanthrene	1.3	10
120-12-7	Anthracene	2.7	10
260-94-6	Acridine	6.1	10
86-74-8	Carbazole	1.9	10
206-44-0	Fluoranthene	3.1	10
129-00-0	Pyrene	1.4	10
56-55-3	Benzo(a)anthracene	2.5	10
218-01-9	Chrysene	2.8	10

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TABLE 3-1

Table of Reporting Limits for Tested Parameters

CAS Number	Compound	Reporting Limit ng/L (PPT)	Reporting Limit ug/L (PPB)
205-99-2	Benzo(b)fluoranthene	2.5	10
207-08-9	Benzo(k)fluoranthene	2.3	10
192-97-2	Benzo(e)pyrene	1.9	10
50-32-8	Benzo(a)pyrene	2.3	10
198-55-0	Perylene	2.5	10
193-39-5	Indeno(1,2,3-cd)pyrene	2.1	10
53-70-3	Dibenz(a,h)anthracene ¹	1.6	10
191-24-2	Benzo(g,h,i)perylene	2.8	10
205-82-3	Benzo(j)fluoranthene ²	-	-
195-19-7	Benzo(c)phenanthrene ³	-	-
215-58-7	Dibenz(a,c)anthracene ¹	1.6	-
192-65-4	Dibenzo(a,e)pyrene ³	-	-
189-64-0	Dibenzo(a,h)pyrene ³	-	-
189-55-9	Dibenzo(a,i)pyrene ³	-	-
57-97-6	7,12-Dimethylbenz(a)anthracene	2.8	-
56-49-5	3-Methylcholanthrene	3.5	-
108-95-2	Phenol	-	10
95-48-7	2-Methylphenol	-	10
106-44-5	4-Methylphenol	-	10
95-57-8	2-Chlorophenol	-	10
88-75-5	2-Nitrophenol	-	10
105-67-9	2,4-Dimethylphenol	-	10
120-83-2	2,4-Dichlorophenol	-	10

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TABLE 3-1

Table of Reporting Limits for Tested Parameters

CAS Number	Compound	Reporting Limit ng/L (PPT)	Reporting Limit ug/L (PPB)
59-50-7	4-Chloro-3-methylphenol	-	10
88-06-2	2,4,6-Trichlorophenol	-	10
95-95-4	2,4,5-Trichlorophenol	-	50
51-28-5	2,4-Dinitrophenol	-	50
100-02-7	4-Nitrophenol	-	50
534-52-1	4,6-Dinitro-2-methylphenol	-	50
87-86-5	Pentachlorophenol	-	50
	Total Phenolics	-	5
1	Dibenz(a,h)anthracene and Dibenz(a,c)anthracene coelute.		
2	Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either benzo(b)fluoranthene or benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistently separated by this method. Therefore, if present, it will be detected and reported as benzo(b) and/or benzo(k)fluoranthene.		
3.	Analytical standards not consistently available. It has not been demonstrated that this component can be routinely detected by this method.		

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varies slightly for the different aquifers (Mt. Simon-Hinckley, Ironton-Galesville, Prairie du Chien-Jordan, St. Peter, and Drift-Platteville) being monitored in accordance with the CD-RAP.

The overall sampling program is summarized in Tables 3-2, 3-3, and 3-4, and Figures 3-1 through 3-4.

3.2 Objectives and Intended Data Usage

Analytical levels for this project incorporate aspects of levels IV, and V, as defined by "Data Quality Objectives for Remedial Response Activities" (U.S. EPA, 1987). Contents of reports and data packages provided by the analytical laboratory will be based on those specified in Contract Laboratory Program (CLP) Statement of Work (SOW) Document OLM01.8, August 1991, (the deliverables are discussed in Section 10.3 in this QAPP). Data validation criteria are derived from "National Functional Guidelines for Organic Data Review" (U.S. EPA, December 1994). The details for quality control data acceptance criteria are discussed in Section 11 and Appendix B (Analytical Standard Operating Procedures (SOPs)). Data use categories include monitoring during implementation, site characterization, and risk assessment. It is the level of concern for low part per trillion concentrations of PAH that specifies a level V analytical level for this project. Level V includes non-conventional parameters, method-specific detection limits, and the modification of existing analytical methods. Rigorous Quality Assurance/Quality Control (QA/QC) to produce data of known quality are part of this program.

The objective of the GAC treatment system monitoring (CD-RAP Section 4.3) is to assess and evaluate the performance of the treatment system. Analytical results for influent and effluent samples will be compared to the drinking water criteria for PAH as established in the CD-RAP. Based on these comparisons, decisions will be made on: 1) system operations (e.g., when the carbon should be replaced), and 2) cessation of the treatment system, if desired, when sufficiently low concentrations of PAH in influent samples are demonstrated.

The objective of monitoring the four existing Mt. Simon-Hinckley Aquifer municipal drinking water wells and any new Mt. Simon-Hinckley Aquifer municipal drinking water wells installed within one mile of well W23, and analyzing for PAH (CD-RAP Section 5.1), is to assure the continued protection of these wells from PAH resulting from activities of Reilly at the Site. The analytical data will be used to make comparisons between the levels of PAH found in the Mt. Simon-Hinckley Aquifer, and the drinking water criteria established in the CD-RAP.

If any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23 (CD-RAP Section 6.2.1), then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the

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TABLE 3-2

Summary of Sampling and Analytical Program

Sample Matrix	Field Parameter	Number of Samples	Laboratory Parameters	Number of Samples	Field Blanks	Field Duplicates	Matrix Spike ¹	Matrix Spike Duplicate ¹	Matrix Total
GAC Treated Water	X	X	PAH (ppt)	4	4	4	4	4	20
			Acid Fraction compounds ²	1	X	1	1	1	4
GAC Feed Water	X	X	PAH (ppt)	1	X	1	1	1	4
Ground Water	pH	67	PAH (ppt)	103	18	18	18	18	175
	temperature		PAH (ppb)	14	4	4	4	4	30
	Specific Conductance		Total Phenols	14	4	4	4	4	30

1 Matrix spike sample/matrix spike duplicate sample shall consist of the same matrix being analyzed. Triple the normal volume when related matrix spike/matrix spike duplicate samples are to be retrieved.

2 Analysis of sample for acid fraction compounds listed in EPA Method 625 shall be in accordance with Contract Laboratory Program Statement of Work Document OLM01.0, or most recent version.

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TABLE 3-3

Sampling Plan GAC Treatment System Monitoring Schedule^a

RAP Section	Sampling Points	Start of Monitoring	Sampling Frequency	Analyses ^b
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH(ppt) ^c
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH(ppt)
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH(ppt)
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds in EPA Test Method 625
<p>a This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1998. Sections 4 and 12 of the RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. The location of the GAC treatment system is shown in Figure 1.</p> <p>b Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds in EPA Test Method 625 are provided in the QAPP. Field blanks will be collected and analyzed at a frequency of one every ten samples or fewer. Treated water will be duplicated at a rate of 100 percent. Feed water duplicate samples will be collected and analyzed at a frequency of one per ten samples.</p> <p>c ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.</p>				

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TABLE 3-4**Sampling Plan Ground Water Monitoring Schedule^a**

Source of Water	RAP Section	Sampling ^b Points	Start of Monitoring	Sampling Frequency	Analyses ^c
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP 17	Date of plan approval	Annually	PAH(ppt) ^d
	5 3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Ironton-Galesville Aquifer	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH(ppt)
Prairie du Chien-Jordan Aquifer	7.3(A)	SLP4	Start of pumping	Semi-annually	PAH(ppt) phenolics
	7.3(B)	W23	Date of plan approval	Semi-annually	PAH(ppb) ^e
	7.3(C)	SLP6, SLP7 or SLP9	Date of plan approval	Annually	PAH(ppt)
	7.3(D)	W405 or W406 ^f , H3, SLP10 or SLP15, SLP14, SLP16, W402 W403, W119	Date of plan approval	Annually	PAH(ppt)
	7.3(E)	SLP5, H6, E3, MTK6, W29, W40, W70	Date of plan approval	Annually	PAH(ppt)
	7.3(F) ^g	W32, SLP8, SLP10, E4	Date of plan approval	Semi-annually	No chemical analyses ^g
	7.4.1 ^h	W48, W401, E2, E7, E13, E15	Date of plan approval	Semi-annually	PAH(ppt)

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TABLE 3-4

Sampling Plan Ground Water Monitoring Schedule^a

St. Peter Aquifer	8.1.3 ¹	SLP3, W14, W24, W33, W122, W129, W133, W408, W409, W410, W411, W412, P116	Date of plan approval	Semi-annually	PAH(ppt)
Drift-Platteville Aquifer	9.1.3 and 9.2.3	W420, W421, W422	Date of plan approval	Quarterly	PAH(ppb) and total phenols
	9.5	W1, W18, W19, W20, W22, W27, W101, W120, W121, W124, W130, W131, W143, W424, W426, W428, W431, W432, W433, W434, W440	Date of plan approval	Semi-annually	PAH(ppt)

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TABLE 3-4

Sampling Plan Ground Water Monitoring Schedule^a

- a This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 1996. Section 12 of the RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring. Field blanks will be collected at a frequency of one for every ten samples or fewer, and one duplicate sample will be collected for every ten samples.
- b Sampling points are located on the maps shown in Figures 3-1 through 3-4. Letter prefixes to well codes are defined as follows:
- | | |
|-----|----------------------------|
| W | 4-inch monitoring well |
| P | monitoring piezometer |
| SLP | St. Louis Park supply well |
| E | Edina supply well |
| H | Hopkins supply well |
| MTK | Minnetonka supply well |
- c Lists of parameters and descriptions of the methods for analysis of PAH, phenolics, and expanded analyses are provided in the QAPP. Water levels will be measured each time samples are collected for analyses, except for those wells which prove to be inaccessible for such measurements.
- d ppt = parts per trillion. This signifies analysis using selected ion monitoring gas chromatography mass spectrometry.
- e ppb = parts per billion. This signifies analysis by the Non-Criteria Method. If analytical results for individual wells are below 20 micrograms per liter (20 ppb) using this method, then the Low-Level Method will be used on subsequent monitoring rounds.
- f W405 = American Hardware Mutual, W406 = Minikahda Golf Course.
- g Water levels will be measured semi-annually at these wells, except for those wells which prove to be inaccessible for such measurements.
- h In accordance with the Gradient Control Modification System, these wells are now sampled semi-annually as opposed to annually.
- i Section 8.1.3 of the Consent Decree-RAP originally specified St. Peter Aquifer monitoring requirements. Monitoring requirements for 1994, and subsequent years are now

CITY OF ST. LOUIS PARK

NON-RESPONSIVE

CITY OF ST. LOUIS PARK

NON-RESPONSIVE

CITY OF ST. LOUIS PARK

NON-RESPONSIVE

FIGURE 3 - 3 LOCATIONS OF ST. PETER AQUIFER WELLS

CITY OF ST. LOUIS PARK



FIGURE 3 - 4 LOCATIONS OF DRIFT/PLATTEVILLE AQUIFER WELLS

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Site. The analytical data would be used to compare the levels of PAH found in potential Iron-ton-Galesville Aquifer drinking water wells to the drinking water criteria established in the CD-RAP.

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to (CD-RAP Section 7.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the CD-RAP (e.g., drinking water criteria for drinking water wells, and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23).

In addition to water quality data generation, water level data will be used for the purpose of determining ground water flow patterns in the Prairie du Chien- Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to (CD-RAP Section 8.1.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site.

Upon its receipt, analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410, and to drinking water criteria established in the CD-RAP. Water level data will be used to evaluate ground water patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells (CD-RAP Section 9.6) is to monitor the distribution of PAH and phenolics in the aquifer, thus evaluating the source and gradient control systems. Ground water analytical data will be used to compare levels of PAH and phenolics in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the CD-RAP for PAH and phenolics. Water level data will be used to evaluate ground water flow patterns in the Drift-Platteville Aquifer.

In addition to the objectives for laboratory analytical data described above, field measurement data will be collected to aid in the ground water sampling procedure. In accordance with MPCA Guidelines (January 1995) field measurements of temperature, pH, and specific conductance will be made for the purpose of determining that a sufficient volume of water has been purged from the well prior to sampling. The objective of those field measurements is to determine when three successive well volumes exhibiting equivalent temperature pH, and specific conductance have been purged from each monitoring well, so that representative samples may be collected.

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The Site Management Plan outlines the scope of work to be performed in order to monitor the ground water in the St. Louis Park, Minnesota, area in accordance with the CD-RAP related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for ground water monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. Although a GAC treatment system has been constructed to treat water from well W23 and the Drift-Platteville Aquifer source control wells prior to its discharge to surface water receivers, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the CD-RAP. Similarly, a GAC treatment system has been constructed to treat water from well SLP4 prior to discharge to the municipal water supply system; however, monitoring of the effluent is not within the scope of work to be performed under this Plan, as the activity is not embodied in the CD-RAP.

The time period covered by this Plan is from January 1, 1998, or the date of its acceptance and approval by the Agencies, whichever is later, to December 31, 1998. The next subsequent Sampling Plan (RAP Section 3.3) will be submitted by October 31, 1998, covering the 1999 calendar year.

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4.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This project is being conducted in accordance with the CD-RAP for the Reilly Tar & Chemical Corporation N.P.L Site in St. Louis Park, Minnesota. The parties to the Consent Decree include Reilly, the City, EPA, MPCA, and MDH. The project organization shown in Figure 4-1 indicates the involvement of the parties to the Consent Decree, as appropriate. The responsibility for the overall QA/QC on the project is ENSR. Both the City and ENSR are responsible for the completion of the monitoring tasks described in this Plan and project QA/QC. The City is assisted in the retrieval and laboratory analysis of water samples by ENSR and QES, respectively. ENSR is responsible for the field sampling QA/QC and will be performing the biannual audit of QES.

ENSR will be responsible for the coordination of all field sample retrieval and Quanterra Environmental Services (QES), with analytical facilities in Arvada, Colorado, will be responsible for the coordination and completion of all laboratory analyses. Responsibilities of the key positions in the organization of QES are described below:

- **Laboratory Project Manager:** The Laboratory Project Manager is ultimately responsible for all laboratory analyses and is the primary point of contact for issues surrounding this Quality Assurance Project Plan (QAPP), resolving technical problems, modifications to SOPs, etc. The Laboratory Project Manager is responsible for the coordination of routine day-to-day project activities including project initiative, status tracking, data review and requests, inquiries and general communication related to the project.
- **Operations Manager:** The Operations Manager is responsible for oversight of preparation and analysis of PAH samples to ensure that project objectives, requirements and QA/QC criteria are met.
- **Laboratory Supervisor:** The Laboratory Supervisor shall be responsible for daily supervision of technicians and analysts for PAH and total phenolics analyses, including sample extraction and preparation.
- **Analyst:** The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by the QAPP.

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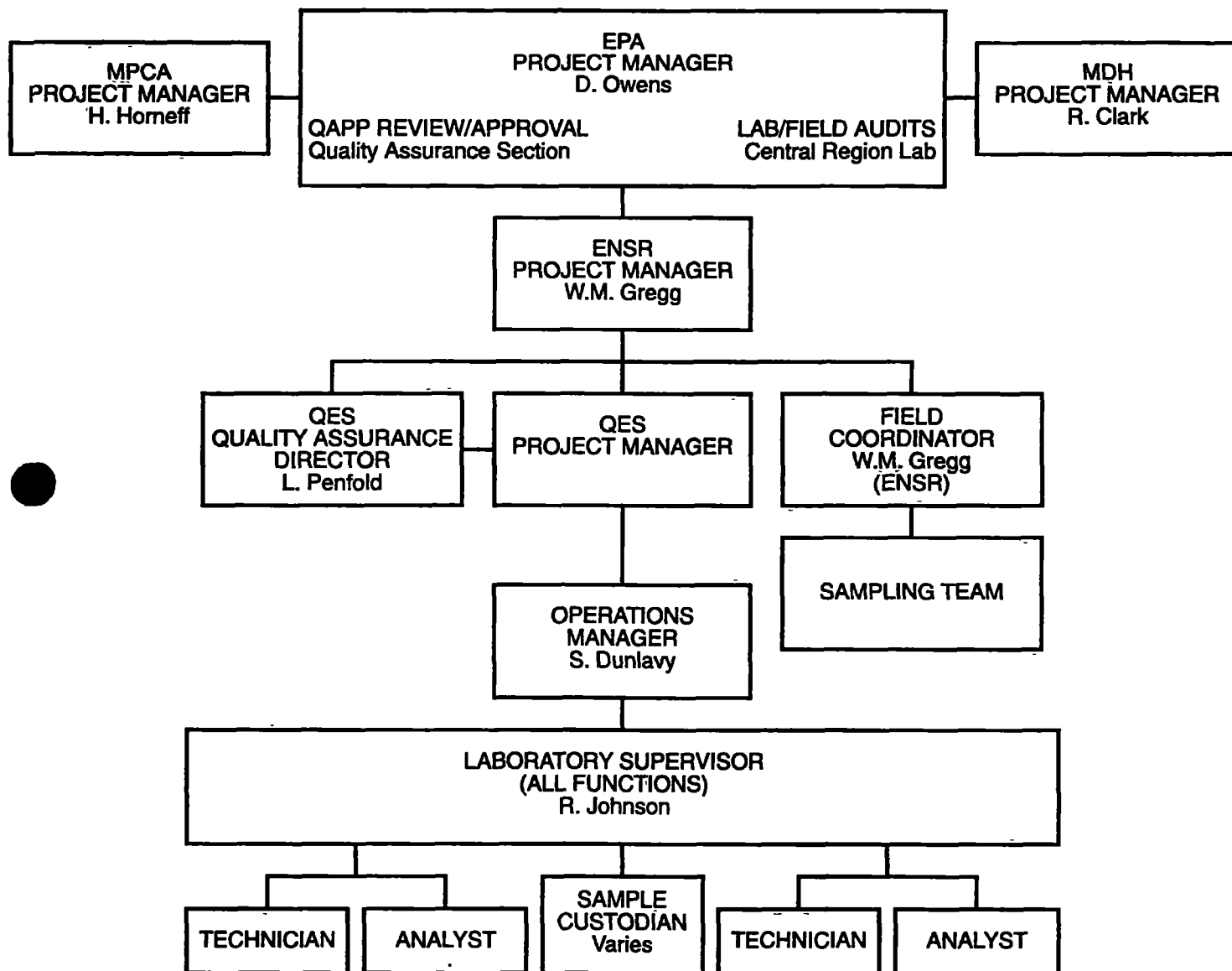


FIGURE 4-1 Program Organization

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- **Technician:** The Technician is responsible for sample extraction. This requires practical experience and knowledge in the techniques of liquid - liquid solvent extraction, Kuderna - Danish evaporation, and the quantitative preparation of sample extracts for analysis.
- **Quality Assurance Director:** The Quality Assurance Director is responsible for overall quality control oversight, including internal audits. The Quality Assurance Director supervises an independent QA/QC department and reports directly to the Division Director and Corporate Vice President for Quality Assurance.
- **Data Assessment:** The evaluation of data, as it is compiled and organized in accordance with the requirements of the QAPP, is the responsibility of the Operations Manager. Additional review, evaluation, and assessment of the data is performed by the Laboratory Manager, thereby providing additional assurance that the requirements of the QAPP are met.

The City's Project Manager shall be responsible to assess the data relative to the objectives and intended data usage identified in Section 3.2. of this QAPP.

The Sampling Team shall consist of employees of the City and ENSR. The team shall be responsible for sample collection, conducting field measurements (i.e. water level), and maintaining proper decontamination procedures stated in the QAPP.

The EPA and MPCA are responsible for review and approval of the Sampling Plan, including the QAPP. In addition, laboratory and field audits may be completed by appropriate EPA representatives. The MPCA is responsible for review of field procedures practiced by the Sampling Team. Responsibilities of the key positions in the EPA and MPCA are described below:

- **EPA Project Manager:** The EPA Project Manager, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA.
- **EPA Quality Assurance Officer:** The EPA Quality Assurance Officer, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA.
- **EPA Central Regional Laboratory:** The EPA Central Regional Laboratory, EPA Region 5, shall be responsible for audits of both field activities and laboratory analyses.

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- **MPCA Project Manager:** The MPCA Project Manager shall be responsible for review and approval of the Sampling Plan, and review of field procedures practiced by the Sampling Team.

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5.0 QUALITY ASSURANCE OBJECTIVES

The principal objectives of the QAPP pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in ground water quality. Therefore, the quality of the data gathered in this project can be defined in terms of the following elements:

- **Completeness** - a sufficient number of successful (valid) measurements to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time. For this project, the completeness objective is that 95 percent of the laboratory analyses and 95% of the field measurements will produce valid data. Field data will be supplemented by resampling if necessary to ensure completeness.
- **Representativeness** - the extent to which reported analytical results truly depict the PAH and phenolics concentrations in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.
- **Accuracy and Precision** - Accurate and precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits. Accuracy and precision will be measured by the analysis of method spikes and duplicate samples.

It is essential that representative ground water samples be retrieved for laboratory analyses. Accuracy and precision in the measurement of parameters used to monitor ground water as it is purged from monitor wells and piezometers will be achieved through the use of standard monitoring procedures carried out continuously during the sample retrieval task. Field measurement equipment will be calibrated in accordance with the manufacturer's recommendations, as outlined in Table 6-6, and appropriate corrective action will be initiated whenever measured accuracy and precision do not meet pre-established limits. Precision and accuracy of field measurement devices will be tested by taking duplicate samples and calculating the relative percent difference using the formula

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presented in Section 14. Duplicate field readings will be completed at a ten percent frequency.

- **Sensitivity** - Determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of one to five times the instrument sensitivity, is used to determine method sensitivity (i.e. method detection limit).
- **Comparability** - the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures.
- **Traceability** - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

1. Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel
2. Quality assessment through a program of regular audits and inspections to supplement continual informal review (refer to Section 12 of this QAPP)
3. Permanent correction of conditions adverse to quality through a closed-loop corrective action system

The City sampling program QAPP has been prepared in direct response to these goals. The QAPP describes the quality assurance program to be implemented and the quality control procedures to be followed by QES during the course of laboratory analyses in support of the various site investigation studies for the City Site. The Quality Assurance objectives will include field blanks, method blanks, field duplicates, surrogate spikes, matrix spikes and matrix spike duplicates. Precision, accuracy and completeness criteria are established for each parameter of

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interest. The specific criteria for each analysis and parameter are set forth in detail in the following sections:

Objective	Frequency (%)	Sections Discussing Criteria
Field Duplicates	10	6.8, 11.1.4
Field Blanks	10	6.5.2
Method Blanks	5	11.1.1, 15.1.3
Surrogate Spikes	100 of GC/MS analyses	11.1.2, 15.1.1
Matrix Spikes/Duplicates	5*	11.1.3, 15.1.2

* One per group of 20 or fewer investigative samples.

6.0 SAMPLING PROCEDURES

Samples will be collected by ENSR and City personnel in accordance with MPCA guidelines (MPCA, 1995). The overall sampling program was summarized in Tables 3-2, 3-3, and 3-4, and Figures 3-1 through 3-4. This section discusses general QAPP provisions relevant to sample collection, containerization, packaging and shipping activities (SOPs 7130 and 7510; Appendix A).

6.1 Training

All ENSR and City personnel working on the project will be properly trained, qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QAPP
- Documentation requirements
- Decontamination requirements
- Health and Safety considerations

Training of field personnel will be provided by the Field Coordinator or a qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed.

6.2 Document Control

Document Control for the Sampling Plan serves a two-fold purpose. It is a formal system of activities that ensures that:

1. All participants in the project are promptly informed of revisions of the QAPP
2. All documents generated during the course of the program are accounted for during, and at the end of the project

This QAPP and all Standard Operating Procedure documents have the following information on each page:

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- Document Number
- Page Number
- Total number of pages in document
- Revision number
- Revision date

When any of these documents are revised, the affected pages are reissued to all personnel listed as document holders with updated revision numbers and dates. Issuance of revisions is accompanied by explicit instructions as to which documents or portions of documents have become obsolete.

Control of, and accounting for documents generated during the course of the project is achieved by assigning the responsibility for document issuance and archiving. Table 6-1 lists the key documentation media for the project and corresponding responsible parties for issuance, execution and archiving.

TABLE 6-1

Document Control

Item	Issued By	Issued To	Archived By
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

6.3 Sample Control Procedures and Chain of Custody

In addition to proper sample collection, preservation, storage and handling, appropriate sample identification procedures and chain of custody are necessary to help insure the validity of the data.

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6.3.1 Sample Identification

Sample labels shall be completed for each sample using waterproof ink. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Time - A 4-digit number indicating the military time of collection.

Sampler - Signature of person collecting the sample.

Remarks - Any pertinent observations or further sample description. The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

XXX-YYYY-~~ZZZZZ~~

XXX	=	Source Code	
		GAC Treatment System	= GAC
		Mt. Simon-Hinckley Aquifer	= MSH
		Ironton-Galesville Aquifer	= IGV
		Prairie du Chien-Jordan Aquifer	= PCJ
		St. Peter Aquifer	= STP
		Drift-Platteville Aquifer	= DPV

YYYYY = Sampling Point Code

~~ZZZZZ~~ = Date Code
Month, day, year

Those samples which will be taken in accordance with this QAPP for quality control purposes will be identified by appending to the sampling point codes the following:

Field blank = FB
Field duplicate = D
Matrix spike = MS
Matrix spike duplicate = MSD

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As an example, a field blank sample taken for the Mt. Simon-Hinckley Aquifer, sampling point SLP11 on January 1, 1991, would be identified as follows:

MSH-SLP11FB-010191

During the sampling event, one sample will be taken per sampling point unless it is duplicated. Duplicate samples will be collected as specified in Table 3-2 (Page 13 of 100). Those samples collected for matrix spike analysis will be selected at the time of sampling and labelled in the field.

After collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

6.3.2 Field Forms

In addition to sample labels and chain-of-custody forms, a field notebook will be maintained by the sample team leader to provide a daily record of significant events. Information to be documented in the notebook will be ground water sample collection records, calibration records, list of samples collected and any other pertinent information such as weather conditions, site visitors, ease/difficulty of retrieving samples, etc. All entries will be signed and dated. All members of the sampling team will use this notebook. The notebook will be kept as a permanent record.

6.4 Sampling Procedures - GAC Treatment System

Chain-of-custody forms will be completed and all samples shipped to QES' laboratory by overnight delivery on the same day they are collected.

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample and matrix spike sample will be collected in six 1-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled.

The GAC treated water samples will have to be collected from two sample taps, one for each column (see Figure 6-1). This will be done by filling three 1-liter bottles from the first column sample tap and then three more bottles from the second (six from each for duplicate samples). No notations distinguishing the two taps will be made on the labels. Only four PAH bottles will be extracted and the extracts composited for analysis.

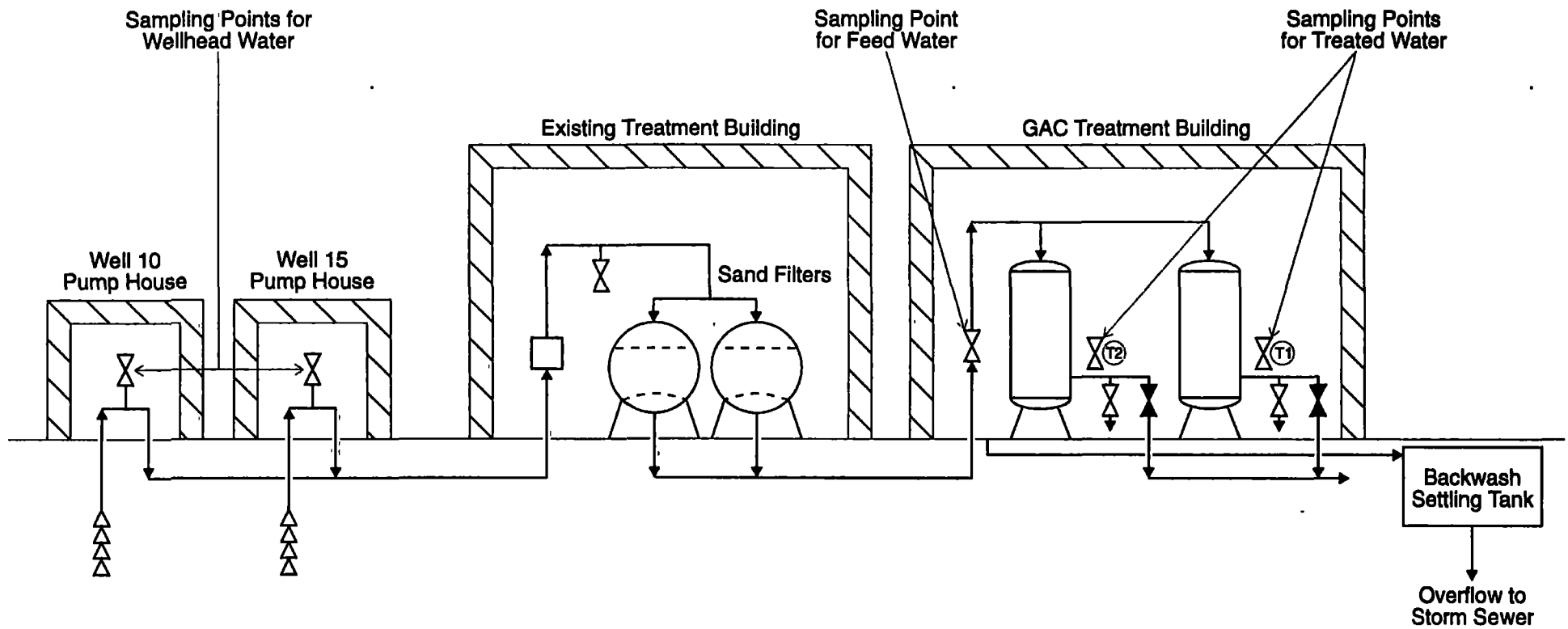


FIGURE 6-1 SAMPLING LOCATIONS

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Field blank samples will be prepared by transferring contaminant-free deionized water provided by QES into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Field duplicate and matrix spike duplicate samples will be obtained by filling 12 one-liter bottles at the sampling point by the procedure described above, splitting these into two groups of six bottles, and assigning a different sample number to each of the resulting six bottle samples. All samples will be packed, cooled to a temperature less than 4°C, and shipped on the day they are collected.

The sampling team must recognize that great care is required to collect samples for part per trillion level PAH analyses that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building for at least 72 hours prior to the day on which PAH samples are to be collected. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed for at least 72 hours prior to the sampling day. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

6.5 Ground Water Sampling and Water Level Measurements

Ground water samples will be collected and water levels measured in accordance with the procedures outlined in this QAPP. The wells involved in the monitoring program include municipal and commercial wells, piezometers and ground water monitoring wells (Table 3-4, Page 15 of 100). Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the ground water at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring points containing progressively higher concentrations of PAH or phenolics.

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6.5.1 Decontamination

The field decontamination procedure to be used on sampling equipment which comes into contact with ground water samples is as follows:

- Disassemble equipment, if applicable
- Wash with Alconox and potable water
- Rinse with potable water

The laboratory decontamination procedure to be used on sampling equipment which comes into contact with ground water samples is as follows:

- Disassemble equipment
- Rinse with methanol
- Scrub with hot soapy water
- Rinse three times with hot deionized water
- Set on aluminum foil, dull side up, air dry
- Bake for one hour at 200°C
- Wrap with aluminum foil, dull side in

6.5.2 Field Blanks

Field blank samples will be prepared by transferring contaminant-free deionized water, provided by QES, into sample bottles in a fashion as closely similar to actual sample collection as possible. This will involve collecting samples through any non-dedicated sample equipment that is decontaminated between samples. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area where samples are being collected at a rate of one per day or where more than ten samples are collected in a day at a rate of one field blank per ten samples.

6.5.3 Sample Containers

For PAH and phenolics, 1-liter amber glass bottles will be used (Table 6-2). Caps will be fitted with pre-cleaned teflon liners. Six bottles are required for each Low-Level PAH sample collected and two bottles for each Non-Criteria PAH and Extended Analysis sample collected. One 16-ounce glass bottle with two milliliters of 50 percent sulfuric acid is required for total phenolics. An independent commercial firm shall provide precleaned bottles to QES for use on this project.

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TABLE 6-2

Sample Containers, Preservation Procedures, and Maximum Holding Times

Parameter	Containers ¹	Preservation ²	Maximum Holding Time ³
Water: PAH (PPT)	Six 1-liter amber glass bottles, Teflon-lined caps	Cool to 4°C, protect from light	Five days (until extraction), 40 days after extraction
PAH (PPB)	Two 1-liter amber glass bottles, Teflon-lined caps	Cool to 4°C, protect from light	Five days (until extraction), 40 days after extraction
Phenolics (Acid Fraction)	Two 1-liter amber glass bottle	Cool, to 4°C	Five days (until extraction), 40 days after extraction
Phenolics (Total)	Two 16-oz. clear glass bottle	Cool to 4°C 2 ml 50 percent H ₂ SO ₄	28 days

Ref: Federal Register Guidelines/Vol. 49, No. 208/Friday, October 26, 1984/ P. 43260

- 1 Matrix spike samples shall consist of the same matrix being analyzed, therefore, triple the normal volume when a related matrix spike sample and matrix spike duplicate are to be retrieved.
- 2 Sample preservation will be performed immediately upon sample collection.
- 3 Samples will be analyzed as soon as possible after validated time of sample receipt (VTSR). The times listed are the maximum times that samples may be held before analysis and still be considered valid.

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In the event QES is required to prepare bottles for sampling, the bottles will be prepared as follows:

1. Wash bottles with hot detergent water.
2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.
3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.
4. Allow to air dry in a contaminant-free area.
5. Caps and liners must be washed and rinsed also. Bottles should be stored and shipped with the Teflon-lined caps securely fastened.

6.5.4 Sample Collection - Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in purging the monitoring wells and piezometers, flexibility in the approach to the method of well purging is necessary. This QAPP proposes that the sampling team be given latitude in the selection of purge equipment/necessary to complete the task (various pumping equipment and procedures that may be used for purging monitoring wells are described in SOP 7130 and MPCA's 1995 Guidelines). In all cases where no dedicated pump exists, samples will be retrieved using laboratory-cleaned, stainless steel or teflon bailers as described below.

Table 3-4 (Page 15 of 100) specifies that Prairie du Chien-Jordan Aquifer monitor well W70, and St. Peter Aquifer monitor wells W24 and W33 be monitored. Each well is equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with monitoring parameters established herein. In the event the dedicated pump within any individual well is inoperable, the pump will be removed, if possible, and purging/sampling procedures will be as established below.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a non-dedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Horiba water quality monitor (or equivalent). Readings will be taken once per well

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volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or teflon bailer and a new length of nylon or polyester rope. All non-dedicated purging and sampling equipment will be decontaminated before use and between sampling points as described in Section 6.5.1 (Page 37 of 100). An equipment blank will be collected at the frequency of one for each ten samples collected from wells that have non-dedicated sampling equipment.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without pre-rinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

6.5.5 Sample Collection - Pumping Wells

At active pumping wells, the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team.

Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. A clean electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered.

If it cannot be determined that a well has been pumping at some time during the 24 hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

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6.6 Sample Preservation, Shipment and Storage

Packaging and shipment of samples shall be in accordance with SOP 7510 (Appendix A). The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAHs are known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples for gas chromatography mass spectrometry (GC/MS) analysis will be extracted within five days of validated time of sample receipt as per CLP SOW Document OLM01.8, or most recent version. The analysis will be completed within 40 days following extraction. The holding time for total phenolics is 28 days from sample collection to analysis.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C ± 2°C. An overnight carrier will be selected to insure delivery at the laboratory within 24-36 hours after collection.

Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C ± 2°C.

6.7 Field Measurement Equipment

All field measurement equipment will be controlled to ensure that measurements obtained are accurate and defensible. Table 6-3 summarizes the parameters to be monitored including calibration and frequency, and quality control criteria (also refer to Appendix A, SOPs 7121, 7123, and 7124).

In addition, these measurement devices will be issued through a formal equipment tracking system and operated by trained personnel.

6.8 Duplicate Samples

Duplicate samples will be collected by alternately filling sample bottles from the source being sampled. For six liter sample collection, one bottle will be filled for the sample, then one bottle for the duplicate, then a second bottle for the sample and then a second bottle for the duplicate, etc. Duplicates will be taken for each analysis type and each sample type, at a rate of one duplicate sample being collected for each ten samples, with a minimum of one duplicate for any sample batch. There are two sample types for this program: GAC treatment system water and ground water.

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TABLE 6-3**Field Measurement Equipment Quality Control**

Parameter	Device	Calibration	Routine Check		Control Limits
			Method	Frequency	
pH	Horiba U-10	Standardize in three or more standard buffer solutions, or use the auto calibration with factory solution	Calibration check-analyze standard buffer solution	1/10 samples	±0.1 pH units
			Analyze duplicates	1/10 samples	±0.1 pH units
			Auto calibration	1/10 samples	±0.1 pH units
Specific Conductance	Horiba U-10	Standardize using two or more KCL solutions or use the auto calibration with factory solution	Calibration check-analyze standard KCL solution	1/10 samples	±1 percent of range being used
			Analyze duplicates	1/10 samples	±1 percent of range being used
			Auto calibration	1/10 samples	±1 percent of range being used
Temperature	Horiba U-10	Factory calibrated	Not required	Not required	±0.1°C

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TABLE 6-3

Field Measurement Equipment Quality Control

				Routine Check	
Depth to Water	Water Level Measurement Device (Electric)	Factory calibrated	Not required	Not required	±0.01 Ft.

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For purposes of fulfilling the ten percent duplicate requirement, all the sampling points shown on Table 3-4 (Page 15 of 100) are the same sample type and have been chosen to maximize the frequency of sample duplication from pumping wells and monitor wells where experience indicates sampling is easiest, thereby insuring consistency of results.

7.0 SAMPLE CUSTODY

The St. Louis Park Ground Water Study is a cooperative effort between the City and ENSR, whose responsibilities include sample retrieval, and QES, whose responsibilities include sample analysis. Proper sample handling and analysis is essential to the success of the study, therefore a formal sample custody procedure has been developed to insure the integrity of all samples. Sections 6.4 and 6.5 discuss field sampling aspects and Section 6.6 outlines procedures for sample preservation, shipment, and storage. This section covers quality related activities from receipt of samples at the QES analytical facilities through issuance of validated analytical data and the storage of data in the final evidence file.

7.1 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures will be followed. A sample is under custody if:

- It is in someone's possession
- It is in someone's view, after being in their possession
- It was in someone's possession and they locked it up to prevent tampering
- It is in a designated secure area

Samples are accompanied by a Chain-of-Custody Record. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst at the laboratory.

Minimum information recorded on the chain-of-custody record, in addition to the signatures and dates of all custodians, will include:

- Sampling site identification
- Sampling date and time
- Identification of sample collector
- Sample identification
- Sample description (type and quantity)

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- Analyses to be performed

Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. Shipping containers will be sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information are entered in the "Remarks" box. Then tear off the last copy of the form and place the original and remaining copies in the container. After the container is closed, place the custody seals on the container.

Whenever samples are split with another laboratory, it is noted in the "Remarks" section. The note indicates with whom the samples are being split and is signed by both the sampler and recipient. If either party refuses a split sample, this will be noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

7.2 Security and Recordkeeping

Samples entering the QES analytical facilities located in Arvada, Colorado, proceed through an orderly chain-of-custody sequence specifically designed to insure continuous integrity of both the sample and documentation.

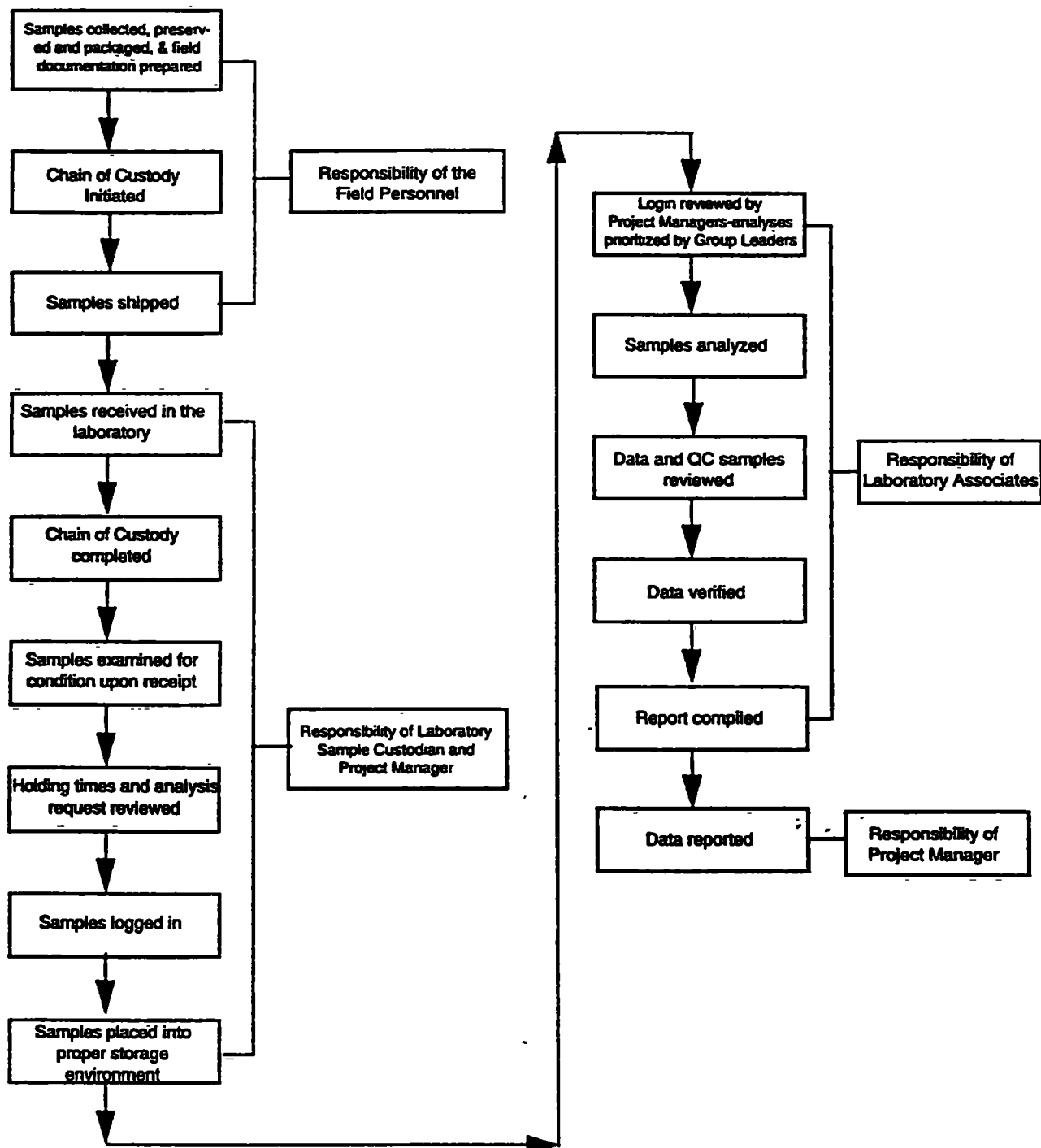
Appendix A contains Standard Operating Procedures (SOPs) which address the following aspects of facility security and sample custody. Figure 7-1 shows the data collection process flow chart.

- Building Security - SOP No. DPOL-QA-002
- Sample Receipt and Chain of Custody - SOP No. DEN-QA-003

7.3 Final Evidence File

The final evidence (or data) files will be maintained for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports raw data, continuing calibration checks, decafluorotriphenyl phosphine (DFTPP) tune, detection limits, chain-of-custody

FIGURE 7-1
Data Collection Process Flow Chart



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documentation, quality control data for blanks and matrix spikes, results forms, and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues is included in the package. The City will maintain these files in a secure, limited access area, under the custody of the Project Manager. QES maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

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8.0 CALIBRATION PROCEDURES

Calibration is required to ensure that field and laboratory analytical systems are operating correctly and functioning at the proper sensitivity to meet established detection limits. For this project, calibration is required for field measurements of temperature, pH, and specific conductance. Appendix A contains the SOPs that describes calibration procedures for field measurement instruments. This project also requires calibration for the four laboratory analyses (Low-Level, Non-Criteria, Extended, and Phenolics). These four analyses are defined in Section 9 of this QAPP.

The laboratory is required to maintain logbooks that contain instrument usage, preventive maintenance, repairs, corrective actions, initial calibrations, daily calibration verifications and calibration standards used.

The specific calibration requirements for each of these analyses are summarized in the subsections below.

8.1 Low-Level (ppt) Analysis

The calibration requirements are described in detail in the SOP for ppt PAH analyses (Appendix B). The discussion below highlights the key aspects of the calibration requirements.

Prior to use of the method for Low-Level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

A midpoint calibration standard is analyzed at the start of each 12-hour calibration sequence and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound listed in Table 8-1 is calculated.

TABLE 8-1

Target Compounds and Key Ions for Low Level PAH Analyses

CAS No.	Compound	Quantitation Mass Ion	Confirmation Ion (Percent Abundance)
271-89-6	2,3-Benzofuran	118	90 (52)

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TABLE 8-1

Target Compounds and Key Ions for Low Level PAH Analyses

CAS No.	Compound	Quantitation Mass Ion	Confirmation Ion (Percent Abundance)
496-11-7	2,3-Dihydroindene	117	118 (57)
95-13-6	1H-Indene	116	115 (108)
91-20-3	Naphthalene	128	102 (7)
4565-32-6	Benzo(B)Thiophene	134	89 (8)
91-22-5	Quinoline ¹	129	102 (20)
120-72-9	1H-Indole	117	90 (31)
91-57-6	2-Methylnaphthalene	141	115 (31)
90-12-0	1-Methylnaphthalene	141	115 (28)
92-52-4	Biphenyl	154	153 (35)
208-96-8	Acenaphthylene	152	151 (17)
83-32-9	Acenaphthene	154	153 (93)
132-64-9	Dibenzofuran	168	139 (40)
86-73-7	Fluorene	166	165 (90)
132-65-0	Dibenzothiophene	184	139 (19)
85-01-8	Phenanthrene	178	176 (19)
120-12-7	Anthracene	178	176 (19)
260-94-6	Acridine	179	178 (26)
86-74-8	Carbazole	167	166 (28)
206-44-0	Fluoranthene	202	200 (17)
129-00-0	Pyrene	202	200 (18)
56-55-3	Benzo(A)Anthracene ¹	228	226 (22)
218-01-9	Chrysene ¹	228	226 (26)

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TABLE 8-1

Target Compounds and Key Ions for Low Level PAH Analyses

CAS No.	Compound	Quantitation Mass Ion	Confirmation Ion (Percent Abundance)
205-99-2	Benzo(B)Fluoranthene ¹	252	250 (22)
207-08-9	Benzo(K)Fluoranthene	252	250 (22)
192-97-2	Benzo(E)Pyrene	252	250 (35)
50-32-8	Benzo(A)Pyrene ¹	252	250 (26)
198-55-0	Perylene	252	250 (24)
193-39-5	Indeno (1,2,3-CD)Pyrene ¹	276	274 (25)
53-70-3	Dibenz(A,H)Anthracene ¹	278	279(20)
191-24-2	Benzo(G,H,I)Perylene ¹	276	274 (25)
205-82-3	Benzo(J)Fluoranthene ¹	252	250 (22)

NOTE: The percent abundance for the confirmation ion is a typical value. Although these ratios will vary, the relative intensities of confirmation ions must agree within plus or minus 20 percent between the calibration standard for any given day and the samples run on that day.

¹ Carcinogenic PAH as defined in Appendix A of the RAP

These daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ± 35 percent of the corresponding calibration curve value, the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, the system is out of control and corrective action must be performed.

The quantitation mass ion, which represents the 100 percent abundance ion, is selected for quantitation and for the daily response factor measurement. The second ion, or confirmation ion, is used for confirmation of the identification. The daily response factor for the quantitation mass ion is compared to the initial calibration curve. During the analysis of the daily calibration standard, the percent abundance of the confirmation ion is obtained. This percent abundance is used for identification purposes for samples analyzed during that day. The percent abundance values shown in Table 8-1 are typical values.

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Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. In the FC43 spectra, the ion abundance of masses 131 and 219 are adjusted to a ratio of 1:1. These two ions are then maximized to be approximately 50 to 70 percent of the ion abundance of the base mass 69. This procedure maximizes the sensitivity of the instrument in the mass region of interest for the PAH analysis.

The requirements above will be employed for all compounds in Table 8-1 with the exception of benzo(j)fluoranthene. Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either Benzo(b)fluoranthene or Benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistently separated by this method. Therefore, if present, it will be detected and reported as Benzo(b) and/or Benzo(k)fluoranthene.

8.2 Non-Criteria Analysis

All Non-Criteria analyses will follow the calibration requirements described in CLP Document OLM01.8, or most recent version. In summary, the SOW requires an initial verification that the mass spectrometer is tuned properly using DFTPP. The SOW also requires an initial five-point calibration be performed for all compounds and that this calibration be verified by the analysis of a daily calibration standard.

The calibration requirements in the SOW are based on the determination of a diverse list of semivolatile organics. Calibration is verified on a daily basis by comparing the responses of a few select compounds, termed calibration check compounds (CCC). Only one of these compounds (acenaphthene) is a target PAH for this project. The response of another group of compounds, termed system performance check compounds (SPCC), are used to verify the analytical system is working properly. None of the SPCCs are target PAH for this project. Finally, the target PAH for this project contain compounds not measured under CLP protocols.

Accordingly, the procedures in the SOW for calibration have been modified to accommodate the differences in the monitoring lists. A calibration standard containing all of the analytes shown in Table 8-1 is used for both initial and continuing calibration in place of the CLP standard. The daily calibration is verified by comparing the response of all 32 compounds to the response from the initial calibrations. For the initial calibration, the relative standard deviation (RSD) for each compound must be less than 30 percent or the system is out of control and corrective action must be performed. For continuing calibration, the percent difference for each compound must be less than 30 percent.

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The control limit for the daily calibration is based on the accuracy and precision objectives of this project and experience with this group of analytes. The limits in the CLP SOW, which is slightly more stringent, is based on a select group of compounds with extensive method performance data.

8.3 Extended Analysis

In addition to the compounds listed in Table 8-1, the compounds shown in Table 8-2 are required to be determined in the extended monitoring program. This extended list of compounds include phenols and other PAHs specified for this project.

TABLE 8-2

Target Compounds for Extended Analyses

CAS No.	A. Other Carcinogenic PAH	Reporting Limit ng/l
195-19-7	Benzo(c)phenanthrene ¹	-
215-58-7	Dibenz(a,c)anthracene ²	1.6
192-65-4	Dibenzo(a,e)pyrene ¹	-
189-64-0	Dibenzo(a,h)pyrene ¹	-
189-55-9	Dibenzo(a,i)pyrene ¹	-
57-97-6	7,12-Dimethylbenz(a)anthracene	2.8
56-49-5	3-Methylcholanthrene	3.5

¹ No analytical standards are available.

² Coelutes with dibenz(a,h)anthracene. If these isomers are detected, they will be reported as a total value.

CAS No.	B. Acidic Compounds Listed in EPA Method 625	Reporting Limit µg/l
108-95-2	Phenol	10
95-57-8	2-Chlorophenol	10
88-75-5	2-Nitrophenol	10
105-67-9	2,4-Dimethylphenol	10
120-83-2	2,4-Dichlorophenol	10
59-50-7	4-Chloro-3-methylphenol	10

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TABLE 8-2

Target Compounds for Extended Analyses

CAS No.	A. Other Carcinogenic PAH	Reporting Limit ng/l
88-06-2	2,4,6-Trichlorophenol	10
51-28-5	2,4-Dinitrophenol	50
100-02-7	4-Nitrophenol	50
534-52-1	4,6-Dinitro-2-methylphenol	50
87-86-5	Pentachlorophenol	50

Analyses for the extended list of compounds will be performed on the semivolatiles extract prepared as described in CLP SOW Document OLM01.8, or most recent version.

Since most of the compounds on the extended monitoring list are also target compounds in the CLP protocol, the CLP calibration protocol will be followed.

The system is tuned with DFTPP and calibrated with the semivolatile compounds as specified in the CLP SOW. The compounds used to assess system performance and to verify the continuing calibration (SPCCs and CCCs) are used to verify that the system is in control. The control limits in the SOW are used. The presence of the PAH compounds listed in Table 8-2 is determined by evaluating the library search results generated for the CLP analysis of the sample.

Example retention times, quantitation ions and the internal standards determined at the laboratory for 7,12-dimethylbenz(a)anthracene and 3-methylcholanthrene are listed in Table 8-3.

TABLE 8-3

Retention Times, Quantitation Ions and Internal Standards for Extended PAH List

Compound	Absolute Retention Time	Relative Retention Time	Quantitation Ions	Internal Standard
7,12-dimethylbenz(a)anthracene	30:51:00 minutes	0.890 minutes	M/Z 256	D ₁₂ -B(A)p ¹ M/Z 264

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TABLE 8-3

**Retention Times, Quantitation Ions and Internal
Standards for Extended PAH List**

Compound	Absolute Retention Time	Relative Retention Time	Quantitation Ions	Internal Standard
3-methylcholanthrene	32:48:50 minutes	1.085 minutes	M/Z 268	D ₁₂ -B(A)p ¹ M/Z 264
1 Benzo(A)Pyrene				

8.4 Phenolics

The calibration requirements are described in detail in the SOP for the total recoverable phenolics analyses (Appendix B). The discussion below highlights the key aspects of the calibration requirements.

A five-point calibration curve covering the linear range of the method will be analyzed prior to the analysis of any samples and with a minimal frequency of once per 12 hours. The calibration curve must have a correlation coefficient of at least 0.995.

An initial calibration verification (ICV) check standard is distilled analyzed at a frequency of one per 20 samples. The measured value from this check standard must be ± 25 percent of the true value.

9.0 ANALYTICAL PROCEDURES

9.1 Low-Level Analysis

As specified in the Consent Decree, four types of analyses are to be performed as part of the RAP for this project. These four analyses are defined below, and the details of the specific analytical procedures are presented in subsequent subsections.

- **Low-Level:** Refers to the determination of a specific list of 21 polynuclear aromatic hydrocarbons using GC/MS with operation in the selected ion monitoring (SIM) mode. The list of target PAH contains carcinogenic and non-carcinogenic compounds and is shown in Table 8-1 of the QAPP. The list includes 14 compounds which are not on EPA's priority pollutant, Appendix IX or Superfund target compound list. The analytical methodology is based on well known principles of GC/MS technology. Although there is no EPA method that embodies this technique for this class of compounds, methods developed for the measurement of polychlorinated dibenzodioxins (e.g., Methods 613 and 8280) are based on selected ion monitoring technology.
- **Non-Criteria:** The Low-Level PAH method is designed to measure PAH at the sub-ppb level. At higher concentrations, the compounds can be measured under scanning GC/MS conditions. Since scanning GC/MS provides more reliable qualitative data, this method, termed "Non-Criteria PAH" is preferred for samples containing ppb concentrations of PAH. The method is based on the Contract Laboratory Program (CLP) protocol for semivolatile organics with the appropriate modifications to address the differences in the monitoring lists.
- **Extended:** Some samples are analyzed for the specific list of compounds shown in Table 8-2 of the QAPP using scanning GC/MS. This list, termed "Extended" analyses, includes additional PAH, specific acid (phenolic) compounds and a provision for "identifying" unknown compounds. Unknown compounds will be identified and reported from the analysis of the acid fraction only. As in the Non-Criteria analyses, analyses are performed using CLP protocols with the appropriate modifications.
- **Phenolics:** Refers to the determination of "total phenols" using a colorimetric procedure.

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A method has been developed for the analysis of selected target PAH and heterocycle compounds at the part per trillion level (ppt, ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by GC/MS in the SIM. The method is generally applicable for the measurement of any PAH or related compound. For this project, only those compounds listed in Table 8-1 will be determined.

In summary, a measured volume of sample is extracted with methylene chloride. Analysis of the concentrated extract is performed by GC/MS using the selected ion monitoring scanning mode under electron impact ionization conditions. Specific details of this methodology can be found in Appendix B, Determination of Low-Level (ppt) PAH and Heterocycles in Water. This method is designed to analyze samples containing up to 600 ppt of an individual PAH. With dilution of the sample extract, the effective range of the method can be extended into the ppb range. However, sample dilutions may result in loss of information concerning recovery of surrogates. For this reason, an optional sample preparation technique is contained in the method. This optional technique can be used if historical information indicates that the target compounds are present in concentrations in excess of 600 ppt.

9.2 Non-Criteria Analysis

The selected target PAH and heterocycle compounds listed in Table 8-1 can be determined by GC/MS in the scanning mode at the ppb and higher concentrations. This analysis, termed Non-Criteria analysis, uses the methodology contained in SOP No. CORP-MS-0001DEN; GC/MS analysis based on Method 8270B, SW-846 (Appendix B). The major deviations to the semi-volatile organic analysis from the SOP are as follows:

1. The calibration is performed as set forth in Section 8.0 of this QAPP
2. The internal QC checks are set forth in Section 11.0 of this QAPP
3. Data are reported only for those compounds listed in Table 8-1

9.3 Extended Analysis

The target compounds listed in Table 8-2 are measured using the methodology contained in CLP SOW Document OLM01.8, or most recent version for semivolatile organics. The only deviations from this SOW are as follows:

1. The calibration is performed as described in Section 8 of the QAPP
2. The only target compound in the analytical reports are those listed in Table 8-2.

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9.4 Phenolics

Total phenolics will be determined by RMAL SOP No. LM-RMA-1112 which references Methods 420.1 and 420.2 as published in the "Methods for Chemical Analysis for Water and Waste, EPA 600/4-79-020" (refer to Appendix B).

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10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 Data Reduction and Validation

All project data will be subjected to a 3-tier process including review by operations, by the data review groups for inorganics and GC/MS and the final review by the Project Coordinator prior to its release. The review process has been developed to minimize errors associated with sample processing, sample analysis and data reporting and to ensure that information pertaining to a given sample is well documented.

Appendix A contains policies and Appendix B contains the SOPs for laboratory data review. Refer to Policy No. QA-012 in Appendix A for information relative to review policies and processes. In addition, the SOPs for the analytical methods in Appendix B contain the calculation techniques required to obtain reportable concentrations from the raw data.

10.2 Turnaround Time

In accordance with Section 3.2 of the RAP, QES has agreed to a 30 working day turnaround. The City, however, makes no enforceable commitment under the RAP except for a maximum of five days from validated time of sample receipt for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, the City makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

10.3 Reporting/Data Deliverables

QES shall prepare summary reports and data packages in a format that mimics the format described in CLP SOW Document OLM01.8, or most recent version. Specifically, Form 1, SV-1 and SV-2 in Exhibit B of the CLP SOW will be changed to include the PAH list of parameters shown in Table 8-1 of the QAPP. Form II, SV-1 will show the surrogates for the PAH analysis. Form III, SV-1 will show the spike compounds for the PAH analyses. Form VI, SV-1 and SV-2 and Form VII, SV-1 and SV-2 will be altered to show just the target parameters shown in Table 8-1 of the QAPP. Finally, Form VIII, SV-1 and SV-2 will be modified to show the internal standards for the PAH method. In addition, in the Low-Level PAH analyses, compounds which are determined to be present in the samples based on careful inspection of the data, but which do not meet the secondary ion confirmation criteria will be flagged with an "R". The reporting forms in Exhibit B will be modified to show the target lists of parameters, surrogates and spiking compounds for the Low-Level PAH.

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The data packages for total phenolics shall as closely follow CLP deliverables for inorganic analysis as possible. Reports shall contain all applicable CLP forms as well as the associated raw analytical data. The package includes Forms I - III, V and VI (results, initial and continuing calibration verification, blanks, matrix spike and duplicate). The report shall be organized as described in CLP Inorganic SOW 7/88.

QES has determined the method detection limits for the ppt PAH analysis of water samples, utilizing GC/MS selected ion monitoring, according to the method described in Appendix B to Part 136 of the Friday, October 26, 1984, Federal Register, Vol. 49, No. 209 - Definition and Procedure for the Determination of the Method Detection - Revision 11.1. Table 10-1 lists the compounds, the observed concentrations of seven replicates spiked at five ppt, the standard deviations and the method detection limits.

QES has also determined the method detection limits for part per billion Phenolics according to Method 420.2 as published in the "Methods for Chemical Analysis for Water and Waste, EPA 600/4-79-020" (see Table 10-2)

TABLE 10-2

Method Detection Limit Study - Total Phenolics

Sample #	Concentration Detected (mg/L)
1	0.0315
2	0.0340
3	0.0291
4	0.0315
5	0.0291
6	0.0291
7	0.0315

Calculated Standard Deviation = 0.0018

Calculated Method Detection Limit = 0.00579 mg/L
= 5.8 µg/l

These calculated method detection limits will be used in sample reporting as follows:

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Quanterra Environmental Services, Denver METHOD DETECTION LIMIT STUDY (Aqueous)

DATE COMPLETED:		10/3/96				PROG/PROJECT:						
METHOD NUMBER:		8270 SIM				PROJECT NUMBER.		48051				
METHOD DESCRIPTION:		PAHs using Select Ion Monitoring				ANALYST:		D. Spence				
PREP METHOD:		3520 (4-Liter extraction)				QUALITY ASSURANCE:		W. Sullivan				
ANALYTE	SPIKE	REPLICATE MEASUREMENT							AVG	Recovery of Spike	PREC.	MDL
	CONC											
		1	2	3	4	5	6	7				
	ng/L								ng/L	%	ng/L	ng/L
2,3-Benzofuran	5	4.41	4.88	4.73	4.20	5.03	4.35	4.59	4.60	91.96%	0.30	0.94
2,3-Dihydroindene	5	6.63	4.82	5.06	4.05	4.76	4.75	4.48	4.94	98.70%	0.81	2.56
1H-Indene	5	5.16	4.65	4.98	4.70	4.96	4.69	4.53	4.81	96.21%	0.23	0.71
Naphthalene	5	6.28	6.27	8.37	6.22	5.98	7.18	6.11	6.49	129.78%	1.01	3.17
Benzo(b)thiophene	5	4.26	4.69	4.46	4.07	4.64	4.26	4.33	4.39	87.76%	0.22	0.70
Quinoline	5	6.76	3.06	4.61	2.58	3.55	3.41	4.81	3.97	79.35%	1.12	3.52
1H-Indole	5	3.41	4.46	3.92	3.45	3.66	3.56	4.12	3.80	75.94%	0.39	1.22
2-Methylnaphthalene	5	6.74	6.29	7.08	6.11	6.00	6.18	5.48	5.98	119.69%	0.63	1.99
1-Methylnaphthalene	5	4.93	6.85	6.50	4.41	5.34	5.12	5.00	5.16	103.28%	0.46	1.44
Biphenyl	5	4.83	6.19	6.40	4.61	5.24	4.84	4.79	4.98	99.69%	0.29	0.91
2,6-Dimethyl naphthalene	5	4.82	6.60	4.89	4.52	4.98	4.84	4.66	4.89	97.71%	0.31	0.98
Acenaphthylene	5	4.56	4.81	4.34	4.24	4.43	4.35	4.36	4.44	88.82%	0.19	0.60
Acenaphthene	5	4.68	4.97	4.39	4.30	4.77	4.61	4.63	4.62	92.42%	0.23	0.71
Dibenzofuran	5	4.92	5.34	4.74	4.55	4.96	4.85	4.74	4.87	97.43%	0.25	0.79
Fluorene	5	6.04	6.14	4.99	4.47	4.71	4.83	4.76	4.85	96.95%	0.23	0.72
Dibenzothiothophene	5	4.73	4.84	4.74	4.52	4.92	4.49	5.20	4.78	95.67%	0.24	0.76
Phenanthrene	5	7.41	7.02	6.86	6.52	6.94	6.60	7.75	7.02	140.30%	0.44	1.38
Anthracene	5	4.73	4.23	4.19	3.55	4.14	3.54	4.44	4.12	82.36%	0.44	1.38

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Quanterra Environmental Services, Denver METHOD DETECTION LIMIT STUDY (Aqueous)

DATE COMPLETED:		10/3/96				PROG/PROJECT:							
METHOD NUMBER:		8270 SIM				PROJECT NUMBER:		48051					
METHOD DESCRIPTION:		PAHs using Select Ion Monitoring				ANALYST:		D. Spence					
PREP METHOD:		3520 (4-Liter extraction)				QUALITY ASSURANCE:		W. Sullivan					
ANALYTE	SPIKE	REPLICATE MEASUREMENT							AVG	Recovery of Spike	PREC.	MDL	
	CONC												
		1	2	3	4	5	6	7					
	ng/L								ng/L	%	ng/L	ng/L	
Acridine	5	4.66	2.71	3.17	1.85	2.19	2.23	3.71	2.93	58.62%	0.99	3.12	
Carbazole	5	5.89	4.55	4.44	4.30	4.76	4.38	5.30	4.80	96.05%	0.58	1.84	
1-Methyl phenanthrene	5	5.26	5.24	4.86	4.85	4.97	4.69	5.66	5.08	101.51%	0.33	1.04	
Fluoranthene	5	6.96	5.72	5.78	5.67	5.77	5.54	6.32	5.97	119.35%	0.50	1.58	
Pyrene	5	6.18	5.38	5.30	5.13	5.24	5.16	5.88	5.47	109.35%	0.40	1.27	
Benzo(a)anthracene	5	4.33	4.01	4.12	4.04	4.10	4.61	4.03	4.18	83.53%	0.22	0.68	
Chrysene	5	4.19	4.32	4.67	5.45	4.37	5.15	4.66	4.69	93.78%	0.46	1.46	
7,12-Dimethylbenz(a)anthracene	5	4.06	3.06	3.45	2.84	3.02	3.19	3.55	3.31	66.19%	0.41	1.29	
Benzo(e)pyrene	5	4.23	4.45	4.46	4.56	4.35	4.87	4.63	4.51	90.12%	0.21	0.65	
Benzo(a)pyrene	5	4.39	3.68	3.84	3.92	3.43	3.56	3.88	3.81	76.25%	0.31	0.98	
Perylene *	5	3.97	3.59	3.67	3.76	3.83	3.79	3.55	3.73	74.70%	0.15	0.50	
Indeno(1,2,3-cd)pyrene	5	4.06	4.89	4.57	4.18	4.36	4.79	4.13	4.43	88.63%	0.33	1.04	
Dibenzo(a,h)anthracene	5	3.94	4.79	4.50	4.08	4.31	4.32	3.81	4.25	85.01%	0.34	1.06	
Benzo(g,h,i)perylene	5	4.46	5.15	5.05	4.48	4.72	6.06	4.81	4.96	99.25%	0.55	1.73	
3-Methylcholanthrene	5	3.27	1.66	1.27	1.28	1.94	0.86	2.17	1.78	35.56%	0.79	2.49	
2,3,6-Trimethyl naphthalene	5	4.97	5.11	4.67	4.31	4.86	4.72	5.02	4.81	96.18%	0.27	0.85	
Benzo(b)fluoranthene	5	4.12	4.47	4.14	3.96	3.97	4.31	3.87	4.12	82.43%	0.21	0.67	
Benzo(k)fluoranthene	5	4.21	4.17	4.70	5.06	4.58	4.97	4.59	4.61	92.19%	0.34	1.07	

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- Analytes detected at concentrations greater than or equal to the calculated method detection limits will be reported with no qualifiers
- Analytes which are not detected will be reported as the calculated detection limit followed by a "U" qualifier which is used in the EPA CLP to indicate a non-detected compound
- Analytes that are detected at concentrations less than the calculated method detection limits will be reported followed by a "J" qualifier which is used in the EPA CLP to indicate that a reported value is below the sample quantitation limit and above the method detection limit

The various items in the data package are listed below:

- Sample Traffic Reports or Chain-of-Custody
- Sample Data Summary Report Including:
 - Case narrative
 - Tabulated target compound results by fraction
 - Surrogate spike analysis results by fraction
 - Matrix spike/matrix spike duplicate results by fraction
 - Blank data by fraction
- Sample Data Package including:
 - Case narrative
 - Traffic reports
 - Raw data

The City will present reports in a manner consistent with the requirements of Section 3.1 of the RAP. In addition, data packages containing all elements listed above will be presented for the sample analyses completed, if so directed by the EPA. The EPA shall be responsible for identifying the specific sample analyses for which data packages will be provided.

10.4 Reporting Requirements for Samples Exceeding Advisory Levels or Drinking Water Criterion

For active drinking water wells, QES will notify the City by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

Parameter	Advisory Level	Drinking Water Criterion
Sum of Benzo(a)pyrene and Dibenzo(a,h)anthracene ¹	3.0 ng/L ¹	5.6 ng/L
Total Carcinogenic PAH ²	15 ng/L ³	28 ng/L ³
Total Other PAH	175 ng/L	280 ng/L

¹ Or the detection limit, whichever is largest

² See Table 10-3

³ Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree

TABLE 10-3

Carcinogenic PAH¹

benz(a)anthracene
benzo(b)fluoranthene
benzo(j)fluoranthene
benzo(ghi)perylene
benzo(a)pyrene²
chrysene
dibenz(a,h)anthracene²
indeno(1,2,3-c,d)pyrene
quinoline

¹ The total maximum levels of carcinogenic PAH established in the Consent Decree-RAP are:

Advisory Level	- 15 ng/l
Drinking Water Criterion	- 28 ng/l

² The total maximum levels of the sum of benzo(a)pyrene and debenz(a,h)anthracene are:

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Advisory Level - 3.0 ng/l (or the lowest concentration that can be quantified, whichever is greater)

Drinking Water Criterion - 5.6 ng/l

Reporting requirements for methods blanks are discussed in Section 11.1.1.

10.5 Final Evidence Files

The final evidence (or data) files will be maintained for the period specified in the RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports, raw data, continuing calibration checks, DFTPP tune, detection limits, chain-of-custody documentation, quality control data for blanks and matrix spikes, results forms, and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues, is included in the package. The City will maintain these files in a secure, limited access area under the custody of the Project Manager. QES maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. This data will be available upon request.

11.0 INTERNAL QUALITY CONTROL

The internal quality control checks will include field blanks, method blanks, surrogate spikes, duplicate analyses, monitoring of internal standard area, and matrix spike analyses. Each quality control check has a specific level of performance which will be reevaluated in an ongoing basis and amended as appropriate through mutual agreement of the EPA, MPCA, and City. The specific details are presented below.

11.1 Low-Level and Non-Criteria PAH Analyses

Internal quality control checks for the Low-Level and Non-Criteria PAH analyses will consist of method blanks analysis, surrogate compound analysis, matrix spike analysis, analysis of duplicate samples, and monitoring of internal standard areas.

11.1.1 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

Method blank analyses are performed at the rate of one per case¹, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

Different control limits have been established relative to method blanks for the Low-Level and Non-Criteria analyses since the target compounds in Table 8-1 are present as "laboratory contaminants" in method blanks at the ppt concentration level.

For the Low-Level analyses, an acceptable method blank analysis must not contain any carcinogenic PAH in Table 8-1 at concentrations greater than or equal to the Method Detection Limits (MDL) in Figure 10-1 or any non-carcinogenic PAH at a concentration greater than five times the MDL. For the Non-Criteria analyses, an acceptable method blank does not contain any PAH in Table 8-1 above ten micrograms per liter. If the method blanks do not meet these

¹ A case is a group or a set of samples collected from a particular site over a given period of time.

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criteria, the analytical system is out of control and the source of the contamination must be investigated and corrective measures taken and documented before further sample analysis proceeds.

11.1.2 Surrogate Compound Analysis

As detailed in the QES SOP (Appendix B), the laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compound will be spiked into the sample prior to extraction to measure individual sample matrix effects associated with sample preparation and analysis.

QES will take corrective action whenever the surrogate recovery is outside the acceptance criteria shown below. The corrective action is described in Section 15 of this QAPP.

In addition, if the recovery of any surrogate is less than 30 percent, the narrative will list the sample together with a comment concerning a possible low bias to the sample result.

Surrogate	Acceptance Criteria %	
	Low-Level	Non-Criteria
Naphthalene-d8	21 - 108	37 - 107
Fluorene-d10	41 - 162	36 - 127
Chrysene-d12	10 - 118	25 - 160

11.1.3 Matrix Spike/Matrix Spike Duplicate Analysis

Low-Level PAH matrix spike and matrix spike duplicate samples will be analyzed as outlined in the QES SOP LM-RMA-3024 (Appendix B). Non-Criteria PAH matrix spike and matrix spike duplicate samples will be analyzed pursuant to applicable criteria in QES SOP CORP-MS-0001DEN.

The laboratory will spike and analyze 5 percent matrix spike and matrix spike duplicate samples. QES will spike seven representative compounds into water. These compounds and the spiking levels are listed below:

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	Low-Level (ng/L)	Non-Criteria (µg/L)
Naphthalene	10	50
Fluorene	10	50
Chrysene	10	50
Indene	10	50
Quinoline	10	50
Benzo(e)pyrene	10	50
2-methylnaphthalene	10	50

The matrix spike criteria for data validity are as follows:

- The Matrix Spike - Matrix Spike Duplicate average for each spike compound must fall between the established acceptable limits.

Matrix Spike Limits

Compound	Low-Level	Non-Criteria
Naphthalene	20 - 150	43 - 128
Fluorene	69 - 118	51 - 120
Chrysene	20 - 132	43 - 124
1H-Indene	20 - 150	49 - 108
Quinoline	20 - 150	40 - 126
Benzo(e)pyrene	20 - 150	20 - 150
2-methylnaphthalene	20 - 150	47 - 138

- Only one compound can be below its required minimum percent recovery. These minimum percent recoveries are:
 1. 10 percent for chrysene and benzo(e)pyrene
 2. 20 percent for all other compounds

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Corrective action will be performed if these criteria are not achieved as described in Section 15.

11.1.4 Duplicates

Relative percent difference between duplicates will be calculated for each detected compound per procedures outlined in Section 14.3 of this QAPP.

11.1.5 Internal Standard Areas

The area of the internal standard will be monitored on each analysis. The area from the daily calibration standard will be used to set a daily acceptance criteria. If the internal standard areas in samples changes by more than a factor of two (-50 percent to + 100 percent) from the daily standard, corrective action must be performed. Additionally, the retention times of internal standards must agree to ± 30 seconds of the daily standards.

11.2 Extended Analysis

The internal quality control checks for Extended Analyses will consist of surrogate spikes, matrix spikes, matrix spike duplicates, method blanks, etc. as described in CLP SOW Document OLM01.8, or most recent version. The acceptance criteria are as defined in the SOW.

11.3 Phenolics

The internal quality control checks for phenolics will mimic those for inorganics in the CLP program and will include the analysis of a method blank, a laboratory check standard, a matrix spike sample, a matrix spike duplicate, and a duplicate sample. The specific details for each of these QC checks are summarized below.

11.3.1 Blanks

A "Preparation Blank" is analyzed with each batch of 20 samples. This blank is carried through the entire procedure, including the distillation step. Additional blanks, termed "Initial Calibration Blank" (ICB) and "Continuing Calibration Blank", (CCB) are also analyzed. These blanks are used only to evaluate the determinative step and are not distilled. They are analyzed at a frequency of one ICB per 20 samples and one CCB per ten samples.

An acceptable blank must not contain phenolics above the nominal reporting limit of five micrograms per liter. If any of the blanks contain phenolics above five micrograms per liter, the system is out of control and corrective action must be performed.

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11.3.2 Laboratory Check Standard

The initial calibration is verified by the analysis of an ICV check standard. A continuing calibration verification (CCV) check standard is analyzed at a frequency of one per ten samples. The measured value for the ICV and CCVs must be within 90 to 110 percent of the true value (these limits are from EPA's Method 420.4, August 1993) for the analytical run to be accepted. If a CCV fails, all the samples analyzed after the last successful CCV must be reanalyzed.

The laboratory uses a phenol standard obtained from a different source than the calibration check standards for the laboratory check standard (LCS). A minimum of one LCS must be analyzed with each batch of prepared samples. The LCS is processed with the samples through all steps of the procedure. The control limits for the LCS are 72 to 115 percent (these limits are statistically based on the laboratory's past performance on the method). If these limits are not met, the associated samples must be reanalyzed.

Quality control charts are used by analysts when trouble-shooting method problems and by the QA office as part of the annual update of historical control limits. The quality control charts are maintained in the QA office.

If the measured values from the check standards are not within control limits, the system is out of control and corrective action must be performed.

11.3.3 Matrix Spikes/Matrix Spike Duplicates

As for the other tests, matrix spikes and matrix spike duplicates will be performed at a frequency of five percent. The spike level is 50 micrograms per liter. The recovery of the matrix spike must be between 75 percent and 125 percent. Corrective action is performed if these criteria are not achieved.

11.3.4 Duplicates

Field duplicate analyses are performed at a frequency of ten percent. Corrective action is performed if the relative difference from the duplicate analysis is greater than 70 percent.

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11.4 Analyst Technical Certification

All analysts must demonstrate their ability to perform any analytical SOP before they are allowed to analyze project samples. This is demonstrated through the successful analysis of two consecutive sets of proficiency QC samples. Each set of proficiency samples consists of two spiked aliquots of a control mix and one method blank, the spike concentrations and acceptance limits are the same as for the LCSs described in the SOP. All proficiency data must be reviewed by the supervisor and a representative of the QA office. The QA manager submits to the department supervisor a written report or certificate of proficiency indicating either need for corrective action or acceptable performance. The proficiency certificate is included in the employee's training file.

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12.0 PERFORMANCE AND SYSTEM AUDITS

The ability of the Sampling Team to successfully monitor pumping wells and monitor wells, and the ability of the laboratory to successfully analyze ground water samples will be confirmed by a series of audits conducted in conjunction with the implementation of the ground water monitoring program established in the CD-RAP.

12.1 Field Audits

EPA Region 5 Central Regional Laboratory (CRL) and the Central District Office (CDO) are responsible for the external audits of field activities, including field sampling and measurements, for compliance of requirements specified for this project. The Quality Assurance Manager and/or Field Team Leader of ENSR will be responsible for internal audits to see if field sampling and measurements are properly followed. Currently, no field audit has been scheduled. Results of any field audit will be forwarded to the EPA and MPCA in accordance with Section 16.

12.2 Laboratory Audits

QES participates in a variety of federal and state certification programs, (including the EPA CLP), that subject the laboratory to stringent systems and performance audits on a regular basis. A system audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data. A performance audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by the auditing agency. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data. Section 15.2.4 discusses audits in more detail. A laboratory audit has been tentatively scheduled for winter of 1996. Results of the audit will be forwarded to EPA and MPCA in accordance with Section 16.

12.2.1 External Audits

QES will be subjected to EPA performance and system audits for approval/ disapproval specific to the requirements of this program. The Laboratory Scientific Support Section (LSSS) of EPA Region 5 CRL is responsible for the audits.

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12.2.2 Internal Audits

In addition to external audits conducted by EPA Region 5 CRL, the City and/or Northwest Regional Quality Assurance Manager of ENSR (office in Fort Collins, Colorado), will be responsible for at least biennial auditing of the QES laboratory. Audit procedures will include both system audits and performance audits as necessary to satisfy the City that QES is capable of rendering satisfactory laboratory services under this QAPP (see form on Page 74 of 100 for the City of St. Louis Park Audit Checklist). Also, ENSR performs its own audit for file completeness and content.

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CITY OF ST. LOUIS PARK AUDIT CHECKLIST

Sample Receiving

YES NO

Are refrigerator/cold storage area temperatures recorded daily and are records properly maintained?

Comments:

Are sample chain-of-custody forms completed properly?

Comments:

Are the temperatures of the coolers being checked and recorded?

Comments

Are volatile samples stored separately?

Comments:

Is access to sample storage area restricted?

Comments:

Data Review

Are all calculations checked by the analyst for accuracy and completeness?

Comments:

Are anomalies documented and reported?

Comments:

What corrective actions are taken when the analytical results fail to meet QC criteria?

Comments:

Standard preparation

Are Class S weights used to check the balances?

Comments:

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CITY OF ST. LOUIS PARK AUDIT CHECKLIST (cont.)

YES NO

Are non-EPA and non-NBS neat materials compared to EPA or NBS whenever possible?

Comments:

Have expired standards and reagents been discarded?

Comments:

Inorganics

Is the conductivity of the Milli-Q water system checked daily and recorded?

Comments:

Is linearity verified (correlation coefficient of at least 0.995) before sample analysis?

If the CCV does not meet acceptance criteria, is the system recalibrated and are all affected samples reanalyzed?

Comments:

Organic Extraction

Are all reagents and solvents screened for potential contamination?

Comments:

What is the source of reagent water?

Comments:

Are spiking solutions and standards prepared from separate stocks?

Comments:

Is glassware cleaned appropriately?

Comments:

Are the hood airflows checked and how often are they checked?

Comments:

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CITY OF ST. LOUIS PARK AUDIT CHECKLIST (cont.)

YES NO

GC/MS Lab

Are current SOP's available for all personnel in the area?

Comments:

Is preventive maintenance performed on all instruments?

Comments:

Have MDL studies been performed on all methods?

Comments:

Are method blanks analyzed with every batch of samples?

Comments:

Are results of QC samples verified to determine if QC criteria has been met before sample analysis begins?

Comments:

Are QC results which are outside of acceptance limits checked for error?

Comments:

Are corrective actions taken as necessary and documented and samples repped/reanalyzed?

Comments:

Are logbooks reviewed periodically, as indicated by the signature/date/comments of the reviewer?

Comments:

13.0 PREVENTIVE MAINTENANCE

Since instrumental methods of analysis require properly maintained and calibrated equipment, the operation and maintenance of modern analytical instrumentation is of primary importance in the production of acceptable data. In order to provide this data, QES subscribes to the following programs:

- Maintenance agreements/service contracts with instrument manufacturers
- Laboratory preventive maintenance program

13.1 Service Contracts

The gas chromatography/mass spectrometry equipment utilized by QES laboratory personnel for this project are covered by maintenance agreements with the instrument manufacturers. These manufacturers provide for both periodic "preventive" service calls as well as the non-routine or emergency calls.

13.2 Instrument Logbooks

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Individual instrument logbooks are maintained for each piece of equipment and located near the instrument. General information contained in the logbooks include:

- Inventory information: Equipment name, model number, serial number, manufacturer, date of acquisition, original cost
- Service tasks and intervals: Cleaning, calibration, operation based on the manufacturer's recommended schedule, and previous laboratory experience
- Service record: Date of breakdown, date of return to service, downtime, problems, repairs, cost of repairs, who performed the repairs, parts required, etc.
- Calibration/performance checks
- Daily operational notes

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Analysts are referred to manufacturers' operating manuals for specific procedures to be followed in the operation and/or maintenance of the individual instruments.

Within each laboratory, a Service Coordinator is assigned the responsibility for overseeing the instrument maintenance program. Group Leaders and analysts actually implement and document the maintenance program.

Each instrument or piece of equipment shall be uniquely identified. Each operating unit shall maintain the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit

The record of maintenance shall include at a minimum:

- Actions taken, including parts replaced
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative

QES documents and describes in detail instrument or equipment preventive maintenance in operation-specific SOPs. SOPs are specific to the type of instrument or equipment being used for sample analysis. Preventive maintenance schedules for instruments used at QES are shown in Tables 13-1 and 13-2.

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TABLE 13-1

Instrument Maintenance Schedule Gas Chromatograph

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ECLD: change roughing resin, clean cell assembly.
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.	Semi-annually ECD: perform wipe test.
Check inlets, septa. When using HP7673 autosampler, change septa daily.	Replace septum (approximately every 100 injections).	Annually ELCD: change finishing resin, clean solvent filter
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller in constant gas flow cannot be maintained.	
	Replace fuse.	
	Reactivate external carrier gas dryers.	

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TABLE 13-1

Instrument Maintenance Schedule Gas Chromatograph

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ECLD: change roughing resin, clean cell assembly.
	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. NPD: clean/replace collector assembly. PID: clean lamp window, replace seals. ECLD: check solvent flow weekly, change reaction tube, replace solvent, change reaction gas, clean/replace Teflon transfer line.	
	Reactivate flow controller filter dryers when presence of moisture is suspected.	
	HP7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.	

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TABLE 13-1

Instrument Maintenance Schedule Gas Chromatograph

Daily	As Needed	Quarterly/Semi- annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ECLD: change roughing resin, clean cell assembly.
	Purge and trap devices: periodic leak checks, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware.	
	Purge and trap autosamplers: leak check system, clean sample lines, valves. PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon valve block, and lubrication of components.	

TABLE 13-2**Instrument Maintenance Schedule
Spectrophotometer**

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero percent T adjustment.	Perform wavelength calibration at 530 nm.	Oil bearings.

13.3 Field Equipment

All field equipment shall be inspected daily for damaged or missing pieces, which will be replaced as needed.

13.3.1 Thermometer

The field worker will handle the thermometer with care to preserve its measurement integrity. After each use, the thermometer will be rinsed with de-ionized or potable water, wiped dry, and returned to its protective case.

13.3.2 Water Level Measurement Tape

Before each use, the battery will be checked using the equipment's element test function, and replaced if necessary. The tape and probe will be wiped clean and rinsed with de-ionized or potable water after each use.

13.3.3 Horiba U-10

The Horiba U-10 shall be maintained in accordance with the manufacturer's requirements. In particular, the battery will be checked daily, and replaced if necessary. The instrument shall be operated and stored at temperatures above freezing, to avoid damaging the instrument. After each use, the instrument will be rinsed with potable or de-ionized water, wiped dry and returned to its storage container.

14.0 SPECIFIC PROCEDURES TO ASSESS DATA PRECISION, ACCURACY AND COMPLETENESS

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to correct, prevent or minimize the recurrence of these events. The quality assessment techniques described below consist of the techniques used to assure that statistical control has been achieved.

The accuracy and precision goals for this project are specified in Section 11. The definitions of accuracy, precision, and completeness, as well as the goal for completeness for this project are discussed in Section 5.

The accuracy and precision of sample measurements are influenced by both external and internal factors. External factors or errors are those associated with field collection and sample transportation. Internal factors or errors are those associated with laboratory analysis. External factors are defined briefly in Section 14.1. Internal factors are defined in Section 14.2.

14.1 External Components

The results for quality control samples taken in the field represent the best estimates of accuracy and precision for the samples, since these values reflect the entire process from samples collection through sample analysis. The frequency of these control samples is described in Sections 5 and 6. Below is a brief description of the information provided by each of these control samples:

- Field blank - provides an estimate of bias based on contamination; includes effects associated with sample preservation, shipping, preparation, and analysis.
- Field collected samples or duplicates - independent samples collected at the same point in space and time. These give the best measurement of precision for sample collection through analysis.

14.2 Internal Components

The results of quality control samples created in the laboratory represent estimates of analysis and precision for the preparation and analysis steps of sample handling. This section describes the quality control-type information provided by each of these analytical measurements. The frequency of each of these measurements is discussed in Sections 5 and/or 11.

- Surrogates - provide an estimate of bias based on recovery of similar compounds, but not the compounds analyzed, for each sample, preparation and analysis.
- Internal standard - an analyte that has the same characteristics as the surrogate, but is added to the sample extract just prior to analysis. It measures bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analysis process only.
- Matrix spikes/Matrix spike duplicates - the matrix spike is added prior to preparation and analysis. The analyte used is the same as that being analyzed and usually is added to a selected few samples in a batch of analyses. It incorporates matrix effects associated with the laboratory analysis.
- Method blanks - provide an estimate of bias based on contamination.

14.3 Calculation Techniques

The quality assessment procedures described above require calculations of relative percent difference (duplicate analyses) and percent recovery (matrix and surrogate spikes). The techniques for performing these calculations are described below.

- Precision - is the degree to which the measurement is reproducible. Precision is assessed by duplicate measurements by calculating the Relative Percent Difference (RPD) between duplicate measurements. The RPD is calculated as follows:

$$RPD = \frac{1D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference
D₁ = first sample value

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D_2 = second sample value (duplicate)

- Accuracy - is a determination of how close the measurement is to the true value.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where: X = the observed value of measurement
T = "true" value

- Completeness - is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under correct normal conditions.

To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. In addition, all data are reviewed in terms of stated goals in order to determine if the database is sufficient.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100\%$$

- Comparability - expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppm, ppb, etc.).

15.0 CORRECTIVE ACTION

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- Blanks contain target analytes above acceptable levels
- Undesirable trends are detected in spike recoveries or RPD between duplicates
- There are unusual changes in detection limits
- Deficiencies are detected by the QA department during internal or external audits or from the results of performance evaluation samples
- Inquiries concerning data quality are received

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, and so on. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA department for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA department.

Generally, out-of-control events or potential out-of-control events are noted on a Laboratory Non-Conformance Memo (NCM) (see Page 87 of 100). The completion of a NCM will follow SOP No. CORP-QA-0010; Non-Conformance and Corrective Action (Appendix A). This form is part of the data package and, thus, must be completed prior to data approval. If an out-of-control event does occur during analysis, for instance, a surrogate recovery falls out the expected range, the analyst must describe on this form: the event, the investigative and corrective action taken, and the cause of the event, and notify the Laboratory Quality Control Director. In some cases, investigation of an out-of-control event will reveal no problems. In such cases, the data are flagged and footnoted on the appropriate forms. The out-of-control event and the investigative action is documented in the narrative. If an out-of-control event is discovered during data

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PROBLEM (Be specific):

PARAMETER _____

QC LOT _____

ANALYST: _____ **DATE:** _____

CORRECTIVE ACTION TAKEN, RESULTS OF ACTION:

ANALYST: _____ **DATE:** _____

SUPERVISOR COMMENTS AND FOLLOW-UP ACTIONS/RESULTS:

SUPERVISOR: _____ **DATE:** _____

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QUALITY ASSURANCE APPROVAL AND COMMENTS:

QA/QC: _____ DATE: _____

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package review, the Laboratory Quality Control Director notifies the supervisor for corrective action.

15.1 Low-Level and Extended PAH Analyses

15.1.1 Surrogates

As discussed in Section 11.1.2, corrective action will be performed whenever the surrogate recovery is outside the following acceptance criteria:

Surrogate	Acceptance Criteria %	
	Low-Level	Non-Criteria
Naphthalene-d8	21 - 108	37 - 107
Fluorene-d10	41 - 162	36 - 127
Chrysene-d12	10 - 118	25 - 160

The following corrective action will be taken when required as stated above:

1. Check calculations to assure there are no errors
2. Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance.
3. If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that an interference specific to the surrogate was present that resulted in the high recovery and this interference would not affect the quantitation of other target compounds. (The presence of this type of interference can be confirmed by evaluating the chromatographic peak shapes and ion intensities of the surrogates.)
4. If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as D with the note surrogate diluted out.

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5. Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis of the extracts yields surrogate spike recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported and documented in the narrative.

15.1.2 Matrix Spikes/Matrix Spike Duplicates

The matrix spike criteria for data validity are as follows:

- The Matrix Spike - Matrix Spike Duplicate average for each spiked compound must fall between the established acceptable limits (refer to Section 11.1.3 for limits).
- Only one compound can be below its required minimum percent recovery.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated or a laboratory control sample (LCS) will be analyzed. If the subsequent matrix spike analysis or the LCS analysis meets the criteria, the data will be considered valid. Both matrix spike and surrogate spike recoveries will be used in assessing QA/QC for QES's analytical work.

TABLE 15-1

Summary of Historical Surrogate Control Recoveries

QC Category	Testcodes	QC Type	Components	Accuracy Limits	Precision Limits
PAHCSLP75A	PAH-CSLP-LL-75-A (Low Level 75)	DCS/LCS/MSSD	Indene	29-105	20
		DCS/LCS/MSSD	Naphthalene	49-123	20
		DCS/LCS/MSSD	Quinoline	29-139	20
		DCS/LCS/MSSD	2-Methylnaphthalene	52-108	20
		DCS/LCS/MSSD	Fluorene	53-105	20
		DCS/LCS/MSSD	Chrysene	32-101	20
			Benzo(e)pyrene	41-121	20
		SCS Blank	Naphthalene-d8	49-102	
		SCS Blank	Fluorene-d10	51-86	

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TABLE 15-1

Summary of Historical Surrogate Control Recoveries

QC Category	Testcodes	QC Type	Components	Accuracy Limits	Precision Limits
		SCS Blank	Chrysene-d12	44-131	
		Sample Surrogates	Naphthalene-d8	24-108	
		Sample Surrogates	Fluorene-d10	21-103	
		Sample Surrogates	Chrysene-d12	1-137	

The values in Table 15-1 are the limits for the surrogate recoveries for the Laboratory Control Samples and Method Blanks. These values were calculated using historical data from prior analyses.

15.1.3 Blanks

If non-carcinogenic PAH are detected in any Low-Level QC method blanks above the MDL but less than five times the MDL, the corrective action will consist of flagging the data and investigating the source of the problem to implement a corrective action for future work. If the concentration of carcinogenic PAH in the method blank exceeds the MDL or the concentration of non-carcinogenic PAH in the method blank exceeds five times the MDL, additional corrective action, including but not limited to, reanalyses of the blank and reanalyses of the samples may be required.

If target compounds are detected in Non-Criteria method blanks above ten micrograms per liter, the corrective action will consist of flagging the data and investigating the source of the problem to implement a corrective action for future work.

The relative concentration of compounds in both the samples and the blank are assessed as part of this corrective action. The results of these activities are documented in the narrative.

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15.2 Other Corrective Actions

These sections discuss corrective actions which will be taken in the event that a sample or sample extract is lost or destroyed during shipment, storage or analysis, or in performance and system audits.

15.2.1 Samples

In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all Low-Level (ppt) samples. For all samples, field blanks and matrix spikes and duplicates, subsequent extraction and analysis will be conducted on four intact 1-liter bottles. All field blank duplicates will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported. Additional sample matrix will be required for matrix spike analyses.

If less than four liters of a sample remains after shipment and storage for analysis, the Program Manager will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

15.2.2 Samples Extracts

If a sample extract is broken or lost during analysis, the Program Manager will be notified and will be responsible for determining the need for replacing the lost sample. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section the action taken.

15.2.3 Quality Control Samples

If a method blank, or matrix spike and its duplicate is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, its duplicate will be analyzed. The analysis report for the sample batch associated with the field blank will clearly note the occurrence in the discussion section.

15.2.4 Audits

Audits of QES are performed to assess the degree of adherence to policies, procedures, and standards. These assessments are conducted internally by QES personnel and externally by clients and regulatory agencies. Audits can identify areas for improvement with regard to compliance with policies, procedures, and standards. Audits also provide a means for correction prior to system failure. The following types of audits and assessments are performed at QES.

- Performance Audits
- Systems Audits
- Data Audits
- Spot Assessments
- Compliance Audits

Internal audits are generally conducted by QA staff, although periodic self-audits may be conducted by the operational units. Audits and assessments are generally conducted through the use of checklists and appropriate reference documents. Systems and compliance audits are conducted with an opening meeting in which representatives from management, key operational staff, and QA staff participate. The opening meeting provides a review of the objectives of the audit and the schedule required to conduct the audit. At the completion of the audit, a debriefing is held to outline the findings, including identification of positive performance, to discuss requirements in areas of deficiencies, and to answer questions. Spot assessments are generally more informal than systems or compliance audits, and may be conducted without prior scheduling.

The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all issues are closed.

15.2.4.1 Performance Audits

Performance audits or performance evaluations are conducted to verify the ability of the laboratory to correctly identify and quantitate compounds in check samples. These samples may be supplied internally or externally as blind or double-blind samples. These samples demonstrate data quality through statistical analysis. The results of internal performance audits may be used to document the training level of the analyst performing the work or to assess the overall performance of the facility. Periodic double-blind performance audits are conducted by QES to assess all aspects of laboratory performance from project initiation through analysis and

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reporting. Each laboratory QA Manager is responsible for ensuring that performance audit sample(s) are analyzed quarterly (either external or internal).

The results of each performance audit shall be reported to laboratory management. All performance audit results which are identified as unacceptable must be investigated. It is recommended that any results which are flagged as exceeding the warning limits, but within the control limits for the study shall also be reviewed. The findings of the investigation and corrective action taken must be documented. This documentation for all external performance audits shall be provided to the agency or client supplying the audit, as well as being included in the QA monthly report to management.

15.2.4.2 Systems Audits

A systems audit assesses fulfillment of the QES Quality Assurance Management Plan (QAMP) and the state of the QES Quality Management System (QMS). Each laboratory undergoes numerous systems audits performed by external parties, including certifying agencies and clients.

15.2.4.2.1 Internal Systems Audits

An annual systems audit will be performed under the direction of the Corporate Director of QA. This audit is performed to assess each laboratory's adherence to the requirements of the QAMP and to assess the status of corrective actions from other audits at that facility.

The Corporate Director of QA shall appoint a lead auditor to conduct the systems audit. A corporate audit checklist shall be used. The lead auditor has the authority to lengthen the audit, revise the scope of the audit, stop work, or specify an accelerated schedule for re-audit. The lead auditor shall be responsible for preparing a report detailing the results of the audit. The report shall be submitted to the audited Laboratory Director and Laboratory QA Manager within four weeks of the audit. Copies of the report shall be distributed to the Regional QA Director, the Regional Operations Vice President, the Senior Vice President of Operations Services, and the President. The audited laboratory must respond in writing within four weeks of receiving the audit report.

The audit report shall have the following sections:

- Introduction
- Purpose
- Scope
- Summary

- Findings
- Comments

Findings are defined as those non-compliant practices which require corrective action. Comments are considered advice and do not require a corrective action response. It is the responsibility of the QA Manager at each facility to verify implementation of the corrective actions and close all internal audit findings. This process shall be documented and the report shall be provided to the recipients of the original audit report.

Internal audit reports shall be maintained according to the QES Record Retention Policy as confidential documents and shall not be released for use outside the laboratory. External auditors may view internal audit reports as part of their on-site audit.

15.2.4.2.2 External Systems Audits

Audits of QES are performed by external agencies and clients. All scheduled audits shall be placed on the facility's calendar with the knowledge of the Laboratory Director and the Laboratory QA Manager to assure no scheduling conflicts occur and that appropriate staff will be available to meet the agencies or client's objectives.

All deficiencies reported to the laboratory must be satisfactorily responded to in a timely manner. Corrective actions taken must also be documented. A copy of the external audit report and the laboratory's response, documenting corrective actions, must be provided to the Laboratory Director, the Regional Director of QA, the Corporate Directory of QA, and the Vice President and General Manager of Laboratory Operations. It is the responsibility of the QA Manager to verify implementation of the corrective actions and close all findings from the audit.

15.2.4.3 Data Audits

Data audits will be routinely performed and documented to ensure that project records meet project requirements as described in method SOPs, project plans, or other documented requirements. The data audit is used to identify any lab errors that may have occurred. The laboratory QA Manager is responsible for performing data audits as specified in QA Policy No. QA-005.

15.2.4.4 Spot Assessments

Spot assessments are conducted to monitor or observe a process or activity in order to verify conformance to the specified requirements for that activity. These assessments are performed monthly, unless a systems audit or follow-up audit is performed by the QA Manager or Corporate QA office. The scope of the assessment is determined by the QA Manager and may be directed based on information obtained from client inquiries, trends in recorded non-conformances, performance audits, or other sources. A spot assessment may be used to assess a procedure performance relative to the documented SOP. This assessment identifies deviations from requirements that may not be detected in a detailed review of the data package alone. Such an assessment is conducted by observation of the associates performing the task compared with the documented SOP. In some cases, the assessment may be conducted through interviews with the associate when observation of a task is not possible. Review of relevant documentation for the completed procedure is included in such an assessment. A checklist may be used in conducting the assessment. The results of the assessment are documented, as are the corrective actions. All deficiencies noted as a result of a spot assessment must be corrected by the responsible staff in a timely manner.

15.2.4.5 Compliance Audits

Compliance audits may consist of any combination of the previously described audits. A compliance audit is conducted to ensure that the laboratory is performing according to explicit contract requirements. These requirements may be stated in a contract, QAPP, Statement of Work, analytical methods, or some combination of these documents. In addition, a compliance audit may include assessment of the administrative requirements of the contract, such as small business subcontracting plans, invoices, and notifications. The technical aspects of the compliance audit are assessed by the QA staff while the administrative aspects are assessed by a representative of the Contract Compliance Officer. Compliance audits are initiated at the request of the Contract Compliance Officer.

15.2.5 Field Audits and Corrective Actions

ENSR oversees field sampling activities including field corrective actions. The only planned field corrective actions are replacement of bottles, bailers, or ground water pumping equipment if these items are damaged in the field. Other corrective actions may be necessary if field meters do not provide measurements within QA/QC limits. Recalibration and maintenance in accordance with manufacturer's specifications is performed, or the meter is replaced. Resampling has occurred in the past to replace samples lost or broken in shipment.

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The location of the monitoring wells is convenient to City and ENSR offices and to stores/vendors where field support is available. Therefore, the sampling programs are always capable of successfully collecting the samples required for the sampling event in accordance with the Sampling Plan.

Data inconsistencies are potentially short-term problems that are addressed by ENSR and the City jointly. During the ten years of CD-RAP monitoring, only one municipal well has contained PAH above advisory levels and the specific course of action to resample that well, in accordance with the CD-RAP was followed. The data have been successfully used for the past ten years to identify "breakthrough" at the carbon treatment plants, and to plan the replacement of the carbon in accordance with the CD-RAP. Other than the resampling prescribed by the CD-RAP, data inconsistencies are not the basis for any field corrective actions. The long-term nature of the ground water containment remediation strategy allows any data inconsistencies to be put into the context of a large data set that defines water quality. The laboratory analytical method has evolved, and has been refined, over the years to avoid data inconsistencies that were apparent during the earlier years of this program. The City and ENSR will continue to evaluate the laboratory analytical procedures in an effort to understand any data inconsistencies and the potential need for corrective actions.

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16.0 QUALITY ASSURANCE REPORTS TO LABORATORY MANAGEMENT

Executing and administering an effective QA program in a large and complex laboratory system demands the skills of a highly qualified staff. The organizational structure of QES's Quality Assurance Group (Figure 16-1) provides a disciplined national management network which oversees and regulates all laboratory QA functions.

QES's Quality and Technology Group is headed by the Vice President of Quality Assurance, who reports directly to the Senior Vice President of Operations. This position is responsible for oversight of a services program which monitors and controls laboratory operations. This involves the intricate process of developing QA manuals, QC protocols, training programs, SOPs, uniform statistical data, interlaboratory and intralaboratory performance evaluation studies, and internal auditing programs. The Vice President of Quality Assurance and Technology is responsible for the administration and implementation of the QA program at all QES laboratories.

Laboratory QA activities are specifically designed to fulfill the requirements of both the individual laboratory and QES. Directing these activities are the Corporate Director of Quality Assurance who works closely with the Regional Quality Assurance Director, who in turn enforces and monitors the program.

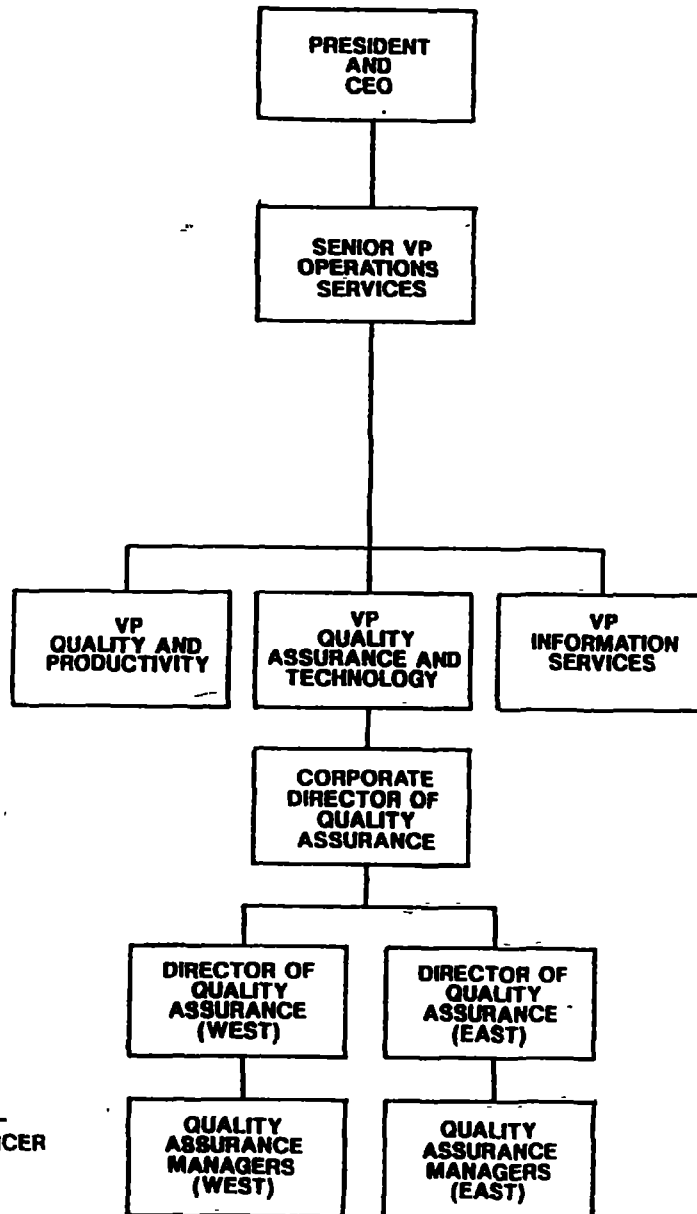
Because a QA program undergoes its most stringent test at the laboratory level, Laboratory QA Managers hold a cornerstone position in the organizational structure. QES QA Managers are highly skilled analytical scientists, knowledgeable in all aspects of laboratory operations. Their responsibilities include diagnosing quality defects and resolving problems with the analytical system; conducting performance evaluation studies, in-house audits, and walk-throughs; performing statistical analyses of data; auditing spike sample results; enforcing chain-of-custody procedures; assisting in the development of QA manual, SOPs and QC protocols; conducting QA training programs; and maintaining extensive records and archives of all QA/QC data.

Laboratory QA personnel report directly through the Regional QA Director and to Corporate Director of Quality Assurance. They also interface with one another in a peer evaluation and auditing system that encourages assistance and feedback, problem analysis, and collaboration on ways to improve laboratory performance.

In conjunction with the Laboratory QA Department, laboratory directors, and managers are responsible for a subset of QA activities, and work closely with supervisors to evaluate daily laboratory functions.

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LEGEND:

CEO = CHIEF EXECUTIVE OFFICER
VP = VICE PRESIDENT

Figure 16-1 Quanterra Quality Assurance Group
Organizational Chart

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The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. Regional QA Directors submit extensive monthly reports to the Vice President of QA and to the Vice President and General Manager of Laboratory Operations. These reports include:

- The results of all systems audits including any corrective actions taken
- Performance evaluation scores and commentaries
- Results of site visits and audits by regulatory agencies and clients;
- Problems encountered and corrective actions taken
- Holding time violations
- Comments and recommendations

The Regional QA Directors submit monthly reports to the Vice President and General Managers of Laboratory Operations. These reports summarize the information gathered through the laboratory reporting system and contain a thorough review and evaluation of laboratory operations throughout QES.

A



APPENDIX A
STANDARD OPERATING PROCEDURES

INDEX OF STANDARD OPERATING PROCEDURES AND POLICIES

SOP NUMBER	SUBJECT	NO. OF PAGES
7121	Field and Laboratory Measurement of pH	8
7123	Field and Laboratory Measurement of Temperature	5
7124	Field and Laboratory Measurement of Specific Conductance	8
7130	Ground Water Sample Collection from Monitoring Wells	19
7510	Packaging and Shipment of Samples	7
DPOL-QA-002	Building Security	6
DEN-QA-0003	Sample Receipt and Chain of Custody	18
CORP-QA-0010	Non-Conformance and Corrective Action	17
POLICY NUMBER	SUBJECT	NO. OF PAGES
QA-012	Technical Data Review Requirements	7

**Field and Laboratory
Measurement of pH****Date:** October 1997**Revision No:** 0**Author:** Lori Fuller**Discipline:** Water**1.0 INTRODUCTION****1.1 Purpose and Applicability**

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of pH meters, including the Hydrolab, Hydac Multimeter Probe, Orion SA 230, YSI Model 3500, and Horiba U-10. Although these meters may measure additional parameters (e.g., temperature, specific conductivity, etc.), this SOP addresses pH measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of pH in accordance with EPA Method 150.1 and Standard Method 4500-H B which address electrometric pH measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

- 2.1 The analyst is responsible for verifying that the pH meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.
- 2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- pH meter
- pH meter manufacturer's instruction manual
- Deionized water
- Clean glass beakers or cups
- 4.0, 7.0, and 10.0 buffer solutions
- Magnetic stirrer and Teflon-coated stirring bar
- Lint-free tissues
- 10% hydrochloric acid
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

- 4.1.1 To achieve accurate pH measurements, samples should be analyzed in the field (preferably within 15 minutes), or as soon as possible after collection. Sample should be collected in plastic or glass containers.
- 4.1.2 After measuring a sample containing oily material or particulate matter, the electrode must be cleaned by carefully wiping with a lint-free cloth, or washing gently in a mild detergent, followed by a deionized water rinse. If this does

not suffice, an additional rinse with 10% hydrochloric acid (followed by deionized water) may be needed.

- 4.1.3** As temperature can affect the pH measurements obtained, both the pH and the temperature of the sample must be recorded.
- 4.1.4** Calibration must include a minimum of two points that bracket the expected pH of the samples to be measured. An example of a calibration sheet is presented in Figure 1.
- 4.1.5** Primary standard buffer salts available from NIST can be purchased and are necessary for situations where extreme accuracy is required. Secondary standard buffers may be purchased as a solution from commercial vendors and are recommended for routine use. Buffers should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year should be used if the manufacturer does not supply an expiration date or if the buffers are prepared from pH powder pillows, etc.
- 4.1.6** When using the meter in the laboratory, always place the buffer/sample beaker on the magnetic stirrer, and make sure the stirring bar is rotating during measurements. Rinse the stirring bar as well as the beaker between buffers/samples. CAUTION: The magnetic stirring plate can generate heat when used for an extended period of time, and lead to increased temperature of the buffers/samples.
- EXCEPTION: Do not use the magnetic stirrer for acid rain samples. It is crucial not to induce dissolved gases into the sample to be absorbed or desorbed, as this will alter the pH. Stir the sample gently for a few seconds after introducing the electrode, then allow the electrode to equilibrate prior to recording temperature and pH readings.
- 4.1.7** When the meter is being used in the field, move the probe in a way that creates sufficient sample movement across the sensor; this insures homogeneity of the sample and suspension of solids. If sufficient movement has occurred, the readings will not drift (<0.1 pH units). Rinse the electrode

with deionized water between samples and wipe gently with a lint-free tissue.

- 4.1.8 When measuring the pH of hot liquids, wait for the liquid to cool to 160°F or below.
- 4.1.9 Fluctuating readings may indicate more frequent instrument calibrations are necessary.
- 4.1.10 A "low sodium error" electrode may be used for samples with a pH greater than 10, to reduce sodium error.

4.2 Calibration and Measurement Procedures

- 4.2.1 The pH meter must be calibrated daily before any analyses are performed. The meter should be recalibrated every 12 hours or at the frequency specified in the project plan.
- 4.2.2 Connect the electrode to the meter. Choose either 7.0 and 10.0 (high range) or 4.0 and 7.0 (low range) buffers, whichever will bracket the expected sample range. Place the buffer in a clean glass beaker. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Measure and record the temperatures of the buffers using a calibrated thermometer or automatic temperature compensation (ATC).
- 4.2.3 Place the electrode into the 10.0 buffer or into the 7.0 buffer.
- 4.2.4 Adjust the instrument calibration according to the manufacturer's instructions. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.
- 4.2.5 Refill the beaker with the 7.0 buffer or the 4.0 buffer. Rinse the electrode, gently wipe it with a lint-free tissue, and place it in the selected buffer solution. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Continue adjusting the instrument calibration according to the manufacturer's instructions. Record the electrode slope (if provided by the instrument) on the calibration sheet (an acceptable slope is between 92 and 102 percent). Measure and record the

temperature of the buffer using a calibrated thermometer or ATC. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

- 4.2.6 An additional check may be performed, if required by the project plan, by placing the electrode into an additional buffer solution. This buffer should be from a different source than the buffers used for the initial calibration. This buffer should read within ± 0.2 pH units of the buffer's true pH value.
- 4.2.7 Verify the calibration every 15 samples and at the end of the day with a buffer solution prepared from a different source than that used for initial calibration. Recalibrate the instrument if the check value varies more than 0.2 pH units from the true value.
- 4.2.8 The electrode will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.
- 4.2.9 Recalibrate the instrument if the buffers do not bracket the pH of the samples.
- 4.2.10 The meter must be recalibrated following any maintenance activities and prior to the next use.

4.3 Troubleshooting Information

If there are any performance problems with any of the pH meters which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

- 4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.
- 4.4.2 The electrode must be stored and maintained according to the manufacturer's instructions.

- 4.4.3** If an instrument with ATC is being used, the device should be checked on a quarterly basis for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

- 5.1** Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ± 0.1 pH units.
- 5.2** The temperature readout of the meter will be checked annually against an NIST-traceable thermometer. If the difference is greater than 0.2°C , the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.
- 5.3** Some regulatory agencies may require the analysis of USEPA Water Supply (WS) or Water Pollution (WP) performance evaluation samples. These performance evaluation samples will be analyzed as required.

6.0 DOCUMENTATION

- 6.1** All pH meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet (Figure 1). pH data may be recorded on the appropriate laboratory or field data sheets or logbooks.
- 6.2** Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:
- Date and time of calibration
 - Signature or initials of person performing the measurement
 - Instrument identification number/model
 - Expiration dates and batch numbers for all buffer solutions
 - Reading for pH 7.0 buffer before and after meter adjustment
 - Reading for pH 4.0 or 10.0 buffer before and after meter adjustment
 - Readings for all continuing calibration checks
 - Temperature of buffers (corrected for any difference with reference thermometer), including units
 - Slope reading (if provided by instrument)

- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and pH of sample (including units and duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform pH measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that pH measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

**Field and Laboratory
Measurement of Temperature****Date: October 1997****Revision No: 0****Author: Lori Fuller****Discipline: Water****1.0 INTRODUCTION****1.1 Purpose and Applicability**

This Standard Operating Procedure (SOP) provides basic instructions for routine measurement of temperature using any high quality mercury-filled thermometer or thermistor with analog or digital read-out device such as the Hydrolab, Hydac Multimeter Probe, Seabird 911 CTD, and Horiba U-10. Multimeter instruments used for temperature measurement may measure additional parameters (e.g., conductivity, pH, etc.). This SOP addresses temperature measurement only (other capabilities are outlined in the appropriate SOP). This SOP is designed specifically for the measurement of temperature in accordance with EPA Method 170.1 and Standard Method 2550 B which address thermometric temperature measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

- 2.1 The analyst is responsible for verifying that the temperature measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.
- 2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Thermometer or thermistor with analog or digital read-out device
- Manufacturer's instruction manual for the instrument
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate temperature measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2 Calibration and Measurement Procedures

- 4.2.1 ENSR-owned temperature measuring devices will, at a minimum, be checked annually as described in Section 5.0. The device will be checked against an NIST-traceable thermometer and the necessary compensation made for the difference in temperature between the two. Rental equipment will be checked by the manufacturer and documentation provided to ENSR. Certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual will be calibrated every 6 months in the manufacturer's laboratory using ITS90 standards. ITS90 is the International

Temperature Scale developed by the Joint Panel of Oceanographic Tables and Standards. Post-cruise calibration records received from the manufacturer may be used to post-calibrate field data.

- 4.2.2 Immerse the thermometer or temperature measuring device into the sample.
- 4.2.3 Swirl and take a reading when the value stabilizes.
- 4.2.4 Record the temperature reading to the nearest 0.5° for a thermometer or 0.1° for digital meter-type instruments. For instruments other than the oceanographic instruments, compensate for any difference with the NIST-traceable thermometer.
- 4.2.5 Temperature measuring devices designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the temperature sensor data. Additional documentation requirements are listed in Section 6.2.
- 4.2.6 Temperature data may be post-calibrated using any of a variety of calibration data including, but not limited to, field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post calibration, and the technique used, will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with any of the meter-type temperature measuring devices, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions. If a performance problem exists with the thermometer, discard the thermometer and replace it.

4.4 Maintenance

Instrument maintenance for meter-type temperature measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

5.1 With the exception of the oceanographic instruments, the temperature measuring devices will, at a minimum, be checked against an NIST-traceable thermometer at the frequency stated in section 4.2.1. This verification procedure will be performed as follows:

- Immerse the thermometer or temperature sensor and the NIST-traceable thermometer into a sample.
- Allow the readings to stabilize.
- Record the readings and document the difference.
- Label the thermometer or temperature sensor with the correction value/adjustment and the date the accuracy check was performed.
- Compensate for the difference when sample measurements are taken.

5.2 Duplicate measurements of a single sample will be performed at the frequency stated in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within $\pm 0.5^{\circ}\text{C}$ or approximately $\pm 1.0^{\circ}\text{F}$.

6.0 DOCUMENTATION

6.1 Records for checking the accuracy of the thermometer or temperature measuring device (where applicable) will include:

- Date
- Thermometer or meter-type temperature measuring device checked
- Reference thermometer number
- Readings for reference thermometer and thermometer being checked
- Adjustment made for difference in readings
- Initials of analyst

6.2 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Thermometer ID # or instrument identification number/model
- Sample identification/station location
- Temperature of sample (including units and duplicate measurements) compensated for any difference with the reference thermometer if applicable
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform temperature measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that temperature measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

**Field and Laboratory
Measurement of Specific
Conductance****Date: October 1997****Revision No: 0****Author: Lori Fuller****Discipline: Water****1.0 INTRODUCTION****1.1 Purpose and Applicability**

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of specific conductance meters, including the Hydrolab, Hydac Multimeter Probe, YSI Model 3500, Coastal Microqual, Horiba U-10, and Seabird 911 CTD. Although these meters may measure additional parameters (e.g., temperature, pH, etc.), this SOP addresses specific conductance measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of specific conductance in accordance with EPA Method 120.1 and Standard Method 2510 B which addresses specific conductance measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

- 2.1 The analyst is responsible for verifying that the specific conductance meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.
- 2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Specific conductance meter
- Specific conductance meter manufacturer's instruction manual
- Deionized water
- Clean glass beakers or plastic cups
- Potassium chloride (KCl) solution, 0.01M, for determination of cell constant (0.5M KCl for saline water measurements)
- KCl standard at concentration that approximates sample concentrations
- Lint-free tissues
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 Specific conductance measurements should be taken soon after sample collection since temperature changes, precipitation reactions, and absorption of carbon from the air can affect the specific conductance. If specific conductance measurements cannot be taken immediately (within 24 hours), samples should be filtered through a 0.45 μ filter, stored at 4°C and analyzed within 28 days.

4.1.2 Report results as specific conductance, μ mhos/cm at 25°C.

- 4.1.3 As temperature can affect the specific conductance measurements obtained, record both the specific conductance and the temperature of the sample.
- 4.1.4 Secondary standards may be purchased as a solution from commercial vendors. These standards should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year should be used if the manufacturer does not supply an expiration date or if the standards are prepared from various salts (e.g., KCl).

4.2 Calibration and Measurement Procedures

- 4.2.1 The specific conductance meter must be calibrated daily (or the calibration checked) before any analyses are performed. However, certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual, which, because of their sensitivity, are only calibrated by the manufacturer (at their specified frequency).
- 4.2.2 Set up the instrument according to the manufacturer's instructions.
- 4.2.3 Rinse the probe with deionized water and dry with a lint-free tissue.
- 4.2.4 Repeat the above procedure for the beakers or cups.
- 4.2.5 Pour a sufficient amount of the KCl standard (preferably at a concentration that approximates the sample concentrations) into the beaker or cup to cover the probe.
- 4.2.6 Immerse the probe in the standard.
- 4.2.7 Record the stabilized specific conductance reading of the standard and the temperature. Adjust the instrument reading (according to the manufacturer's instructions) to display the correct value of the standard. If the meter cannot be adjusted to display the correct value of the standard, the standard should read within 5% of the true value. If the meter reading is between 5% and 15% of the true value, calculate the cell

constant using the formula below and correct all subsequent meter readings.

**Cell Constant = 0.01M or 0.5M KCl Standard Conductance
Conductance Meter Reading**

If the meter reading exceeds the reference standard by greater than 15%, replace the instrument. If the meter does not have automatic temperature compensation (ATC), correct all measurements to 25°C by adding 2% of the reading per degree if the temperature is below 25°C and by subtracting 2% of the reading per degree if the temperature is above 25°C.

- 4.2.8 An additional check may be performed, if required by the project plan, by placing the probe into an additional KCl standard. This standard should be from a different source than the standard used for the initial calibration. This standard should read within 5% of the true value.
- 4.2.9 Verify the calibration every 15 samples and at the end of the day. Recalibrate or replace the instrument if the check value is not within 15% of the true value.
- 4.2.10 The probe will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.
- 4.2.11 The meter must be recalibrated following any maintenance activities and prior to the next use.
- 4.2.12 Conductivity meters designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the DO sensor data. Additional documentation requirements are listed in Section 6.0.

4.2.13 Conductivity data may be post calibrated using any of a variety of calibration data including, but not limited to field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post-calibration, and the technique used will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with any of the specific conductance meters which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The probe must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the meter should be checked annually for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 The meter must be calibrated daily before and recalibrated every 12 hours, and will not be used for sample determinations of specific conductance unless the initial check standard value is within 5% of the true value.

5.2 Oceanographic instruments will be calibrated every 6 months in the manufacturer's laboratory using the functional relationship between salinity and conductance, temperature and pressure as defined by the Practical Salinity Scale of 1978 (PSS-78). Post-cruise calibration records received from the manufacturer will be used to post calibrate field data.

- 5.3 Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within 10%.
- 5.4 The temperature readout of the meter will be checked against an NIST-traceable thermometer at least quarterly. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.
- 5.5 Some agencies may require the analysis of USEPA Water Pollution (WP) performance evaluation samples. These performance evaluation samples will be analyzed as required.

6.0 DOCUMENTATION

- 6.1 All specific conductance meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet (an example is presented as Figure 1). Specific conductivity data may be recorded on the appropriate laboratory or field data sheets or logbooks.
- 6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:
- Date and time of calibration
 - Signature or initials of person performing the measurement
 - Instrument identification number/model
 - Expiration dates and batch numbers for all standards
 - Reading for standard before and after meter adjustment
 - Readings for all continuing calibration checks
 - Temperature of standards (corrected for any difference with reference thermometer)
 - Cell constant value
 - Comments
- 6.3 Documentation for recorded data must include a minimum of the following:
- Date and time of analysis
 - Signature or initials of person performing the measurement

- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and conductance of sample (including units and duplicate measurements) Note: show all calculations for converting instrument reading to $\mu\text{mhos/cm}$ if the instrument provides readings in any other units. Useful conversions are: $1 \text{ mS/m} = 10 \mu\text{mho/cm}$ or $1 \mu\text{mho/cm} = 0.1 \text{ mS/m}$.
- Comments.

7.0 TRAINING/QUALIFICATIONS

To properly perform specific conductance measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that specific conductance measurements be taken in the field by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

DO measurements must also be corrected for barometric pressure. DO measurements must also be corrected for barometric pressure. DO measurements must also be corrected for barometric pressure.

Revisión 0



STANDARD OPERATING PROCEDURE

Number: 7130

Date of Issue: 2nd Qtr.1993

Revision: 2

Title: Ground-Water Sample Collection from
Monitoring Wells

Organizational Acceptance

	Authorization	Date
Originator	Christopher Carlo	3/13/84
Technical Reviewer	Arthur Lazarus	3/13/84
Technical Reviewer	Elaine Moore	3/13/84
Technical Reviewer		
Quality Assurance	Scott Whittemore	3/13/84

Revision #	Changes	Authorization	Date
1	• Addition of Equipment Checklists	Scott Whittemore	9/5/86
		Charles Martin	9/11/86
	• The use of electronic sounding devices for water-level measurements has been removed	Elaine Moore	9/10/86
	• Unnecessary steps have been deleted from decontamination procedures		
	• Volume requirements for ground-water purging has been changed from 4 - 10 to 3 - 10 volumes		
	• Additional bailing details added		
	• Additional figures were added		
2	• Miscellaneous rewording		
	• Ground-Water Sample Collection Record, Chain-of-Custody and Sample Label form updates	Mike Dobrowolski	4/27/93
	• Format update		

Organizational acceptance signatures are maintained on file with the original document in the Quality Assurance Library in Acton, MA.

**Ground-Water Sample Collection
from Monitoring Wells****Date: 2nd Qtr. 1993****Revision No: 2****Author: Christopher Carlio****Discipline: Geosciences****1.0 PURPOSE AND APPLICABILITY**

This standard operating procedures (SOP) is concerned with the collection of valid and representative samples from ground-water monitoring wells. The scope of this document is limited to field operations and protocols applicable during ground-water sample collection.

2.0 RESPONSIBILITIES

The site coordinator or designee will have the responsibility to oversee and ensure that all ground-water sampling is performed in accordance with the project-specific sampling program and this SOP. In addition, the site coordinator must ensure that all field workers are fully apprised of this SOP. The field team is responsible for proper sample handling as specified in SOP 7510, Packaging and Shipment of Samples.

3.0 REQUIRED MATERIALS

The list below identifies the types of equipment which may be used for a range of ground water-sampling applications. From this list, a project-specific equipment list will be selected based upon project objectives, the depth to ground-water, purge volumes, analytical parameters and well construction. The types of sampling equipment are as follows:

- **Purging/Sample Collection**

- Bailers
 - Centrifugal Pump
 - Submersible Pump
 - Peristaltic Pump

- **Sample Preparation/Field Measurement**

- pH Meter
 - Specific Conductance Meter
 - Filtration Apparatus

Water-Level Measurement Equipment

Additional equipment to support sample collection and provide baseline worker safety will be required to some extent for each sampling task. The additional material are separated into two primary groups: general equipment which is reusable for several samplings, and materials which are expendable.

- General

Project-specific Sampling Plan

Deionized-water dispenser bottle

Decontamination Solvent-dispenser bottle

Site-specific Health & Safety equipment (gloves, respirators, goggles)

Field data sheets and/or log book

Preservation solutions

Sample containers

Buckets and intermediate containers

Coolers

First-Aid kits

- Expendable Materials

Bailer Cord

Respirator Cartridges

Gloves

Water Filters

Chemical-free paper towels

Plastic sheets

Equipment checklists have been developed to aid in field trip organization and should be used in preparation for each trip.

- ENSR SOP 7131. Field Filtration of Water Samples for Inorganics
- ENSR SOP 7510, Packaging and Shipment of Samples
- ENSR SOP 7600, Decontamination of Equipment

4.0 METHOD

4.1 Water-Level Measurement

- 4.1.1** Prior to obtaining a water-level measurement, cut a slit in one side of a plastic sheet and slip it over and around the well, creating a clean surface onto which the sampling equipment can be positioned. This clean working area should be a minimum of eight feet square. Care will be taken not to kick, transfer, drop, or in any way let soil or other materials fall onto this sheet unless it comes from inside the well. Do not place meters, tools, equipment, etc. on the sheet unless they have been decontaminated.
- 4.1.2** Unlock and/or open the monitoring well. Enter a description of condition of the security system and protective casing on the Ground-Water Sample Collection Record shown in Figure 1.
- 4.1.3** Check for the measuring point for the well. The measuring point location should be clearly marked on the outermost casing or identified in previous sample collection records. If no measuring point can be determined, a measuring point should be established. Typically, the top (highest point) of the protective or outermost well casing will be used as the measuring point. The measuring point location should be described on the Ground-Water Sample Collection Record and should be the same point used for all subsequent sampling efforts.
- 4.1.4** To obtain a water-level measurement lower a decontaminated steel or fiberglass tape into the monitoring well. Care must be taken to assure that the water-level measurement device hangs freely in the monitoring well and is not adhering to the wall of the well casing. The water-level measuring tape will be lowered into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to hundredth of a foot) by repeatedly raising and lowering the tape to converge on the exact measurement. The water-level measurement as well as the point of measurement should be entered on the Ground-Water Sample Collection Record.

4.1.5 Decontamination

The measurement device shall be decontaminated prior to and immediately after use in accordance with ENSR SOP 7600. Decontamination of Equipment. Generally, only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface.

4.2 Purge-Volume Computation.

All monitoring wells to be purged prior to sample collection. Depending upon the ease of purging, 3 to 10 volumes of ground water present in a well shall be withdrawn prior to sample collection or one volume if the well can be purged dry. The volume of water present in each well shall be computed based on the length of water column and well casing diameter. The water volume shall be computed using the volume factors or graph presented in Figure 2.

4.3 Well Purging

Purging must be performed for all ground-water monitoring wells prior to sample collection in order to remove stagnant water from within the well casing and ensure that a representative sample is obtained. The following sections explain the proper procedures for purging and collecting water samples from monitoring wells.

Three general types of equipment are used for well purging: bailers, surface pumps, or down-well submersible pumps.

In all cases pH and/or specific conductance will be monitored during purging. Field parameter values will be entered on the Ground-Water Sample Collection Record along with the corresponding purge volume

4.3.1 Bailing

In many cases bailing is the most convenient method for well purging. Bailers are constructed using a variety of material, generally, PVC stainless steel, and Teflon®. Care must be taken to select a specific type of bailer that suits a study's particular needs. Teflon® bailers are generally most "inert" and are used most frequently. Keep in mind the diameter of

each monitoring well so that the correct size bailers are taken to the site. It is preferable to use one bailer per well; however, field decontamination is a relatively simple task if required.

Bailing presents two potential problems with well purging. First, increased suspended solids may be present in samples as a result of the turbulence caused by raising and lowering the bailer through the water column. High solids concentrations may require that total suspended solids (TDS) and the chemical character of solids be evaluated during sample analyses. Second, bailing may not be feasible for wells which require that greater than twenty (20) gallons be removed during purging. Such bailing conditions mandate that long periods be spent during purging and sample collection or that centrifugal pumps be used. All ground-water collected from monitoring wells for subsequent volatile organic compounds analyses shall be collected using bailers, regardless of the purge method.

4.3.2 Surface Pumping

Ground-water withdrawal using pumps located at the ground surface is commonly performed with centrifugal or peristaltic pumps.

All applications of surface pumping will be governed by the depth to the ground-water surface. Peristaltic and centrifugal pumps are limited to conditions where ground water need only be raised through approximately 20 feet of vertical distance. The lift potential of a surface pumping system will depend upon the net positive suction head of the pump and the friction losses associated with the particular suction line, as well as the relative percentage of suspended particulates.

Surface pumping can be used for many applications of well purging and ground-water sample collection. In all cases, pumping cannot be used for the collection of samples to be analyzed for volatile organic compounds (VOCs).

- **Peristaltic Pump**

Peristaltic pumps provide a low rate of flow typically in the range of 0.02-0.2 gallons/min (75-750 ml/min). For this reason, peristaltic pumps are not particularly effective for well purging. Peristaltic pumps are suitable for purging situations where disturbance of the water column must be kept minimal for particularly sensitive analyses.

Peristaltic pumps are most often used in conjunction with field filtering of samples and therefore can be used to obtain water samples for direct filtration at the wellhead.

- **Centrifugal Pump**

Centrifugal pumps are designed to provide a high rate of pumping, in the range of 10-40 gallons per minute (gpm), depending on pump capacity. Discharge rates can also be regulated somewhat provided the pump has an adjustable throttle.

When centrifugal pumps are used, samples should be obtained from the suction (influent) line during pumping by an entrapment scheme (Figure 3). Construction of this sampling scheme is relatively simple and will not be explained as part of this SOP. It is suggested that if samples cannot be obtained before going through the pump, that samples be obtained by using a bailer once pumping has ceased. Collecting samples from the pump discharge is not recommended.

4.3.3 Submersible Pump

Submersible pumps provide an effective means for well purging and in some cases sample collection.

Submersible pumps are particularly useful for situations where the depth to water table is greater than twenty (20-30) feet and the depth or diameter of the well requires that a large purge volume be removed during purging.

ENSR uses the Johnson-Keck pump model SP-81 which has a 1.75 inch diameter pump unit. The pump diameter restricts use to monitoring wells which have inside

diameters equal to or greater than two (2) inches. As with other pump-type purge/sample collection methods, submersible pumps will not be used for the collection of samples for analyses of volatile organic compounds. Submersible pumps should never be used for well development as this will seriously damage the pump.

4.4 Sample Collection Procedures

4.4.1 Bailing

Obtain a clean/decontaminated bailer and a spool of polypropylene rope or equivalent bailer cord. Using the cord at the end of the spool, tie a bowline knot or equivalent through the bailer loop. Test the knot for security and the bailer itself to ensure that all parts are intact prior to inserting the bailer into the well.

Remove the protective foil wrapping from the bailer, and lower the bailer to the bottom of the monitoring well and cut the cord at a proper length. Bailer rope should never touch the ground surface at any time during the purge routine.

Raise the bailer by grasping a section of cord using each hand alternately in a "rocking" action. This method requires that the samplers' hands be kept approximately 2-3 feet apart and that the bailer rope is alternately looped onto or off each hand as the bailer is raised and lowered.

Bailed ground water is poured from the bailer into a graduated bucket to measure the purged water volume.

For slowly recharging wells, the bailer is generally lowered to the bottom of the monitoring well and withdrawn slowly through the entire water column. Rapidly recharging wells should be purged by varying the level of bailer insertion to ensure that all stagnant water is removed. The water column should be allowed to recover to 70-90% of its static volume prior to collecting a sample. Water samples should be obtained from midpoint or lower within the water column.

Samples collected by bailing will be poured directly into sample containers from bailers which are full of fresh ground water. During sample collection, bailers will not be allowed to contact the sample containers.

4.4.2 Peristaltic Pump

Place a new suction and discharge line to the peristaltic pump. Silicon tubing must be used through the pump head. A second type of tubing may be attached to the silicon tubing to create the suction and discharge lines. Such connection is advantageous for the purpose of reducing tubing costs, but can only be done if airtight connections can be made. Tygon tubing will not be used when performing well purging or collecting samples for organic analysis. The suction line must be long enough to extend to the static ground-water surface and reach further should drawdown occur during pumping.

Measure the length of the suction line and lower it down the monitoring well until the end is in the upper 2-5 inches of the water column present in the well. Start the pump and direct the discharge into a graduated bucket.

Measure the pumping rate in gallons per minute by recording the time required to fill a selected volume of a bucket. Flow measurement shall be performed three times to obtain an average rate.

The pumping shall be monitored to assure continuous discharge. If drawdown causes the discharge to stop, the suction line will be lowered very slowly further down into the well until pumping restarts.

Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record.

Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and specific conductance) have stabilized.

When the sample bottles are prepared, each shall be filled directly from the discharge line of the peristaltic pump. Care will be taken to keep the pump discharge line from contacting the sample bottles. Ground-water samples requiring filtration prior to placement in sample containers, will be placed in intermediate containers for subsequent filtration or filtered directly using the peristaltic pump.

At each monitoring point when use of the peristaltic pump is complete, all tubing including the suction line, pump head and discharge line must be disposed of. In some cases where sampling will be performed frequently at the same point, the peristaltic pump tubing may be retained between each use in a clean zip-lock plastic bag.

4.4.3 Centrifugal Pump

- Direct Connection Method (Note: This method requires that the well casing be threaded at the top.)

Establish direct connection to the top of the monitoring well if possible using pipe connections, extensions, and elbows, with Teflon® tape wrapping on all threaded connections. If the centrifugal pump will subsequently be used for sample collection, a sample isolation chamber will be placed in the suction line configuration as shown in Figure 3.

Prime the pump by adding tap water to the pump housing until the housing begins to overflow.

Start the pump and direct the discharge into a graduated bucket or a bucket of known capacity (>2.5 gallons).

Start the pump and measure the pumping rate in gallons per minute by recording the time required to fill the graduated bucket. Flow measurement should be checked periodically to determine if pumping rates are continuous, fluctuating, or diminishing. If discharge stops, the pump will be throttled back to determine if pumping will restart at a lower rate. If pumping does not restart, the pump should be shut off to allow the well to recharge.

Measurements of pH and specific conductance will be made periodically during well purging. All readings will be entered on the Ground-Water Sample Collection Record. Samples will be collected after the required purge volume has been withdrawn and the field parameters (pH and specific conductance) have stabilized. Samples should be collected from an in-line discharge valve or with a bailer. The pump should be properly decontaminated between wells.

- Down-Well Suction-Line Method

Lower a new suction line into the well. The suction line will have a total length great enough to extend to the water table and account for a minimum of five (5) feet of drawdown. Note should be made that drawdown may exceed the depth where pumping will terminate as a result of a limitation derived from suction-line conditions and the lift potential of the pump. All connections should be made using Teflon® ferrules and Teflon® thread wrapping tape. Run the pump as per Section 4.4.3.

At each monitoring well when use of a centrifugal pump is complete, all suction line tubing should be disposed of properly.

4.4.4 Submersible Pump

Prior to using a submersible pump, a check will be made of well diameter and alignment. A 1.75 inch diameter decontaminated cylindrical tube should be lowered to the bottom of each monitoring well to determine if the alignment or plumbness of a well is adequate to accommodate the submersible pump. All observations will be entered in the Ground-Water Sample Collection Record.

Slowly lower the submersible pump into the monitoring well taking notice of any roughness or restrictions within the riser.

Count the graduations on the pump discharge line and stop lowering when the stainless steel portion is below the uppermost section of the static water column within monitoring

well. Secure the discharge line and power cord to the well casing.

Connect the power cord to the power source (i.e., rechargeable battery pack or auto battery) and turn the pump on (forward mode). When running, the pump can usually be heard by listening near the well head.

Voltage and amperage meter readings on the pump discharge must be checked continuously. The voltage reading will decline slowly during the course of a field day representing the use of power from the battery. Amperage readings will vary depending upon the depth to water table. Amperage readings greater than 10 amps usually indicate a high solids content in the ground water which may cause pump clogging and serious damage. If a steady increase in amperage is observed, the pump should be shut off, allowed to stop, switched to the reverse mode, stopped again and then placed in forward mode. If high amperage readings persist, the pump should be withdrawn and checked using the large upright cylinder and tap water. Ground-water conditions such as high solids may require that an alternate purge/sample method be used

Drawdown must also be monitored continuously by remaining near the well at all times and listening to the pump. When drawdown occurs, a metallic rotary sound will be heard as the pump intake becomes exposed and ceases to discharge water, but continues to run. The pump should be lowered immediately to continue pumping water within the uppermost section of the static water column.

NOTE: The submersible pump cannot be allowed to run while not pumping water for more than five seconds or the pump motor will burn out.

If drawdown continues to the extent that the well is pumped dry, the pump should be shut off and the well allowed to recharge. This on/off cycle may need to be repeated several times in order to purge the well properly.

Measurements of the pumping rate, pH, and specific conductance should be made periodically during well purging.

All readings and respective purge volumes should be entered on the Ground-Water Sample Collection Record.

While pumping is on-going and when sample bottles are prepared, bottles will be filled directly from the discharge line of the pump taking care not to touch sample bottles to the discharge line.

At each monitoring well when use of the submersible pump is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures contained ENSR SOP 7600 Decontamination of Equipment.

4.5 Sample Preparation

4.5.1 Prior to sample collection or shipment, ground-water samples may require filtration and/or preservation dependent on the specific type of analysis required.

4.5.2 Specific preservation techniques are described in the EPA document, Handbook for Sampling and Sample Preservation of Water and Wastewater (EPA-600/4-82-029). The EPA manual and laboratory manager should be consulted during the planning stage of the project. Project-specific sampling plans shall be assembled using the approved procedures obtained from the EPA manual

4.5.3 Filtration

Ground-water samples collected for dissolved metals analyses will be filtered prior to being placed in sample containers in accordance with ENSR SOP 7131, Field Filtration of Water Samples for Inorganics.

Ground-water filtration will be performed using a peristaltic pump and a 0.45 micron, water filter. Typically the water filters are 142 mm in diameter and are usually placed in 142 mm polycarbonate housings.

The filtration of ground-water samples shall be performed either directly from the monitoring well or from intermediate sample containers such as decontaminated buckets. In either

case, well purging shall be performed first. Fresh ground water shall then be filtered and discharged from the filtration apparatus directly into sample containers.

For most dissolved metal analyses, pH adjustment of the sample is also required and shall be performed after filling the sample bottles. This is generally accomplished using laboratory supplied compounds such as sulfuric or nitric acid and sodium hydroxide. The preservative shall be identified in the Quality Assurance or Sampling Plan.

5.0 QUALITY CONTROL

Quality control requirements depend on project-specific circumstances and objectives and should be addressed in the Quality Assurance Project Plan (QAPP).

6.0 DOCUMENTATION

A number of different documents must be completed and maintained as a part of ground-water sampling effort. The documents provide a summary of the sample-collection procedures and conditions, shipment method, the analyses requested and the custody history. The list of documents is:

- Ground-water sample collection record
- Sample labels
- Chain of custody forms and tape
- Shipping receipts

Sample labels shall be completed at the time each sample is collected and will include the information listed below. A sample label is shown in Figure 4.

- Client or project name
- Sample number
- Designation (i.e., identification of sample point no.)
- Analysis

- Preservative (e.g., filtration, acidified pH<2 HNO₃)
- Sample-collection date
- Sampler's name

Figure 5 displays the chain of custody record used by ENSR. The chain of custody form is the record of sample collection and transfer of custody. Information such as the sample collection date and time of collection, sample identification and origination, client or project name shall be entered on each chain of custody record. In accordance with 40 CFR 261.4(d) the following information must accompany all ground water samples which are known to be non-hazardous and to which U.S. Department of Transportation and U.S. Post Office regulations do not apply. Such information is:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of each sample,
- date of shipment, and
- description of sample.

The chain of custody forms provide a location for entry of the above-listed information.

7.0 REFERENCES

EPA, Handbook for Sampling and Sample Preservation of Water and Wastewater
EPA-600/4-82-029, September 1982.

Geotrans, Inc. RCRA Permit Writer's Manual, Ground-Water Protection prepared
for U.S. EPA. Contract No. 68-01-6464, October 1983.

Code of Federal Regulations, Chapter 40 (Section 261.4(d)).



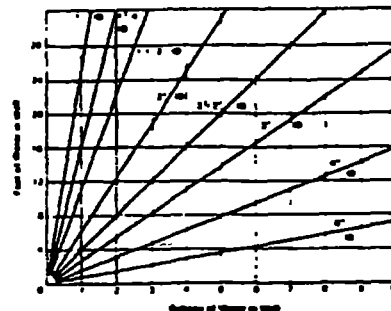
WELL NO.

GROUND WATER SAMPLE COLLECTION RECORD

Project No. _____ Date _____ Time: Start _____ am/pm
Project Name _____ Finish _____ am/pm
Location _____
Weather Conds.: _____ Collector _____

1. WATER LEVEL DATA: (measured from ToC)

- a Total Well Length _____ Well Casing Type _____
- b Water Table Depth _____ Casing Diameter _____
- c Length of Water Column _____ (a-b)
- d Calculated Purgeable Volume _____



2. WELL PURGEABLE DATA

- a. Purge Method _____
- b. Required Purge Volume (@ _____ well volumes) _____
- c. Field Testing Equipment Used _____

Volume Removed	T°	PH	Spec. Cond.	Color	Other
----------------	----	----	-------------	-------	-------

3. SAMPLE COLLECTION:

Method

Container Type

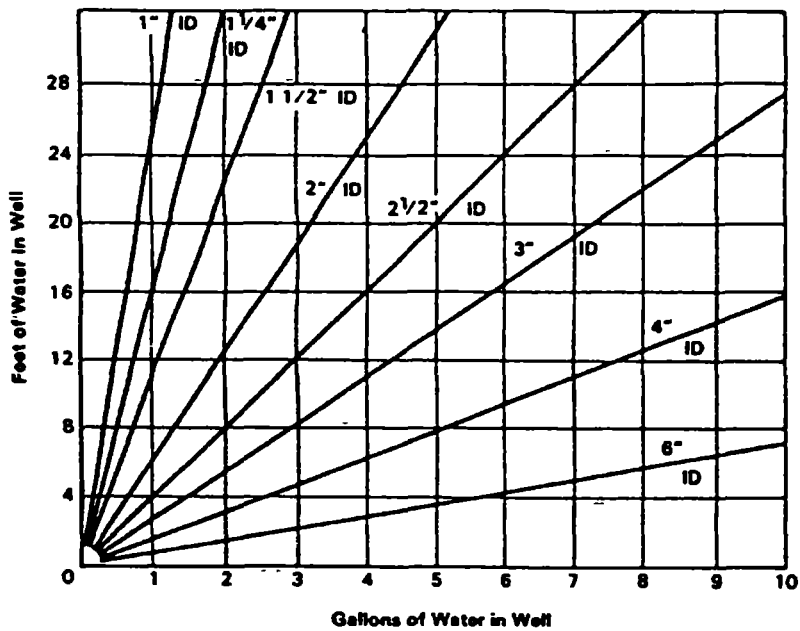
Preservation

Analysis Req

Comments

Abstract

Figure 1

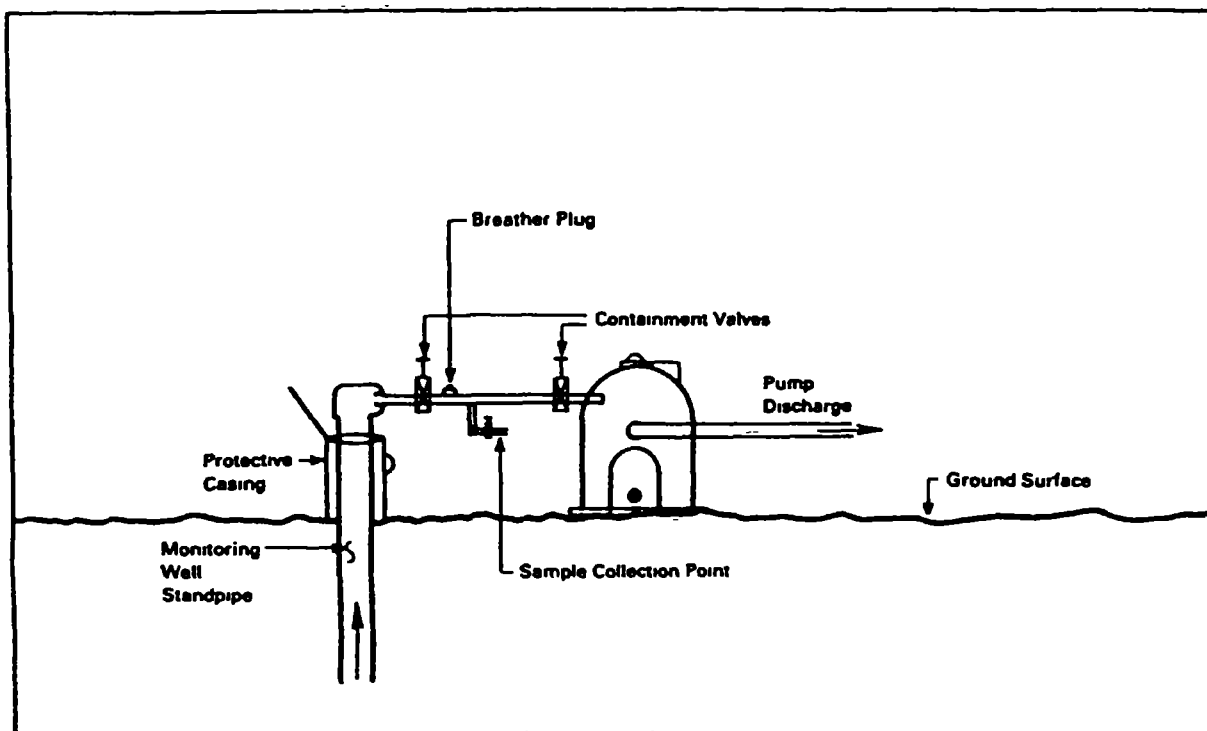


(a) Graphical Explanation

Volume/Linear Ft. of Pipe		
ID(in)	Gal	Liter
1/4	0.003	0.010
3/8	0.006	0.022
1/2	0.010	0.038
3/4	0.023	0.087
1	0.041	0.154
2	0.163	0.618
3	0.367	1.39
4	0.653	2.47
6	1.47	5.56

(b) Volume Factors

Figure 2 Purge Volume Computation



9-02003

Figure 3 Down Well Suction Line Configuration

ENSR		M010271
ENSR Consulting and Engineering		
SITE _____	PROJECT# _____	
SAMPLE ID# _____		
ANALYSIS _____		
PRESERVATIVE HNO ₃ , H ₂ SO ₄ , OTHER _____		
DATE _____	TIME _____	
SAMPLER _____		
OTHER _____		

Figure 4 Sample Container Label

Figure 5 Sample Chain-of-Custody Record

Organizational acceptance signatures are maintained on file with the original document in the Quality Assurance Library in Acton, MA.

Packaging and Shipment of Samples**Date: 2nd Qtr. 1993****Revision No: 2****Author: Christopher Carlo****Discipline: Geosciences****1.0 PURPOSE AND APPLICABILITY**

This Standard Operating Procedure (SOP) describes the procedures associated with the packaging and shipment of samples. Two general categories of samples exist: environmental samples consisting of air, water and soil; and waste samples which include non-hazardous solid wastes and hazardous wastes as defined by 40 CFR Part 261.

2.0 RESPONSIBILITIES**2.1 Project Manager**

It is the responsibility of the project manager to assure that the proper packaging and shipping techniques are utilized for each project.

2.2 Field Team Leader

The field team leader shall be responsible for the enactment and completion of the packaging and shipping requirements outlined in the project specific sampling plan. The field team leader shall be responsible to research, identify and follow all applicable U.S. Department of Transportation (DOT) regulations regarding shipment of materials classified as waste.

3.0 REQUIRED MATERIALS

- Sample cooler
- Bubble wrap
- "Blue Ice" refreezable ice packs
- Fiber tape

- Zip lock plastic bags

4.0 METHOD

The objective of sample packaging and shipping protocol is to identify standard procedures which will minimize the potential for sample spillage or leakage and maintain field sampling program compliance with U.S. EPA and U.S. DOT regulations.

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when all of the following conditions are applicable:

- Samples are being transported to a laboratory for analysis;
- Samples are being transported to the collector from the laboratory after analysis;
- Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

Qualification for transportation as described above require that sample collectors comply with U.S. DOT and U.S. Postal Service (USPS) regulations. If U.S. DOT and USPS regulations are found not to apply, the following information must accompany all samples and will be entered on a sample specific basis on chain of custody records:

- sample collector's name, mailing address and telephone number,
- analytical laboratory's name, mailing address and telephone number,
- quantity of sample,
- date of shipment,
- description of sample, and

In addition, all samples must be packaged so that they do not leak, spill or vaporize.

- 4.1 Place plastic bubble wrap matting over the base and bottom corners of each cooler or shipping container as needed to manifest each sample.
- 4.2 Obtain a chain of custody record as shown in Figure 1 and enter all the appropriate information as discussed above. Chain of custody records will include complete information for each sample. One or more chain of custody records shall be completed for each cooler or shipping container as needed to manifest each sample.
- 4.3 Wrap each sample bottle individually and place standing upright on the base of the appropriate cooler, taking care to leave room for some packing material and ice or equivalent. Rubber bands or tape should be used to secure wrapping, completely around each sample bottle.
- 4.4 Place additional bubble wrap and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler.
- 4.5 Place ice or cold packs in heavy duty zip-lock type plastic bags, close the bags, and distribute such packages over the top of the samples. Add additional bubble wrap/styrofoam pellets or other packing materials to fill the balance of the cooler or container.
- 4.6 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain of custody form. Sign and date the chain of custody tape.
- 4.7 To complete the chain of custody form enter the type of analysis required for each sample, by container, under the "ANALYSES" section. Under the specific analysis enter the quantity/volume of sample collected for each corresponding analysis.
- 4.8 If shipping the samples where travel by air or other public transportation is to be undertaken, sign the chain of custody record thereby relinquishing custody of the samples. Relinquishing custody should only be performed when directly transmitting custody to a receiving party or when transmitting to a shipper for subsequent receipt by the analytical laboratory. Shippers should not be asked to sign chain of custody records.

- 4.9 Remove the last copy from the chain of custody record and retain with other field notes. Place the original and remaining copies in a zip-lock type plastic bag and place the bag on the top of the contents within the cooler or shipping container.
- 4.10 Close the top or lid of the cooler or shipping container and with another person rotate/shake the container to verify that the contents are packed so that they do not move. Improve the packaging if needed and reclose.
- 4.11 Place the chain of custody tape at two different locations on the cooler or container lid and overlap with transparent packaging tape. For coolers with hinged covers, if the hinges are attached with screws, chain of custody tape should also be used on the hinge side.
- 4.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of two full wraps of packaging tape will be placed at least two places on the cooler. Shake the cooler again to verify that the sample containers are well packed.
- 4.13 When transporting samples by automobile to the laboratory, and where periodic changes of ice are required, the cooler should only be temporarily closed so that reopening is simple. In these cases, chain of custody will be maintained by the person transporting the sample and chain of custody tape need not be used. If the cooler is to be left unattended, then chain of custody procedures should be enacted.
- 4.14 If shipment is required, transport the cooler to an overnight express package terminal or arrange for pickup. Obtain copies of all shipment records as provided by the shipper.
- 4.15 If the samples are to travel as luggage, check with regular baggage.
- 4.16 Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and will sign "received by laboratory" on each chain of custody form. The laboratory will verify that the chain of custody tape has not been broken previously and that the chain of custody tape number corresponds with the number on the chain of custody record. The analytical laboratory will then forward the back copy of the chain of custody record to the sample collector to indicate that sample transmittal is complete.

5.0 QUALITY CONTROL

Not Applicable

6.0 DOCUMENTATION

As discussed in Section 4.0 the documentation for supporting the sample packaging and shipping will consist of chain of custody records and shipper's records. In addition a description of sample packaging procedures will be written in the Field Log Book. All documentation will be retained in the project files following project completion.

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ENSR

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Sig. _____

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SOP No. DPOL-QA-002
Revision No. 0
Revision Date: 8/28/97
Page: 1 of 6

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: BUILDING SECURITY

(SUPERSEDES: LP-RMA-0001, REVISION 0)

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Approved by: *Jeff Mercer* 9/30/97
Environmental Health and Safety Coordinator, Jeff Mercer

Approved by: *Thomas Daniels* 9/19/97
Laboratory Director, Thomas Daniels

Proprietary Information Statement:

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1. PURPOSE

- 1.1. The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.
- 1.2. Quanterra's security policy includes controlled access to the building, testing areas, and data files. This policy also extends to include the confidentiality agreements with all personnel, identification badges for all personnel, electronic security and fire alarm systems. Also, all visitors to Quanterra are required to wear visitor badges and should be accompanied by a Quanterra employee during their stay in the facility.

2. RESPONSIBILITIES

- 2.1. It is the responsibility of all employees to maintain the confidentiality of every client's data.
- 2.2. The Human Resources Coordinator (HR Coordinator) is responsible for issuing employee identification badges and keeping track of signed "Confidentiality Agreements". These documents are kept in individual Personnel Files.
- 2.3. The Receptionist is responsible for issuing visitor badges during normal business hours. The Receptionist is also responsible for ensuring that all visitors sign-in and sign-out of the visitor logbook.
- 2.4. Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

3. SAFETY

- 3.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.
- 3.2. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

4. PROCEDURE

- 4.1. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Building Security
 - 4.2.1. All exterior doors to the facility will remain locked at all times with the exception of the front entrance.
 - 4.2.2. During the hours of 8:00 a.m. to 5:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entry ways. The alarm system is not activated during this period.
- 4.3. Personnel Identification
 - 4.3.1. All employees and visitors are required to wear security badges at all times while on the premises of all Quanterra divisions.
 - 4.3.2. The HR Coordinator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.
 - 4.3.3. The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by a Quanterra employee before access to any building will be allowed.
- 4.4. Building Alarm System

Each employee will receive a personal security code and security training at the time of their orientation, which is provided by the Health and Safety Officer and/or the Lab Manager. The procedure is confidential information and can only be obtained from the Health and Safety Officer and/or the Lab Manager.

5. DEFINITIONS

None.

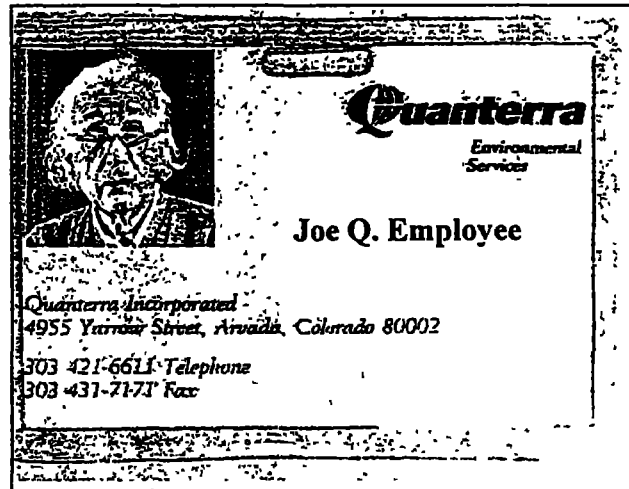
6. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

6.1. Appendix I: Example Employee ID Badge

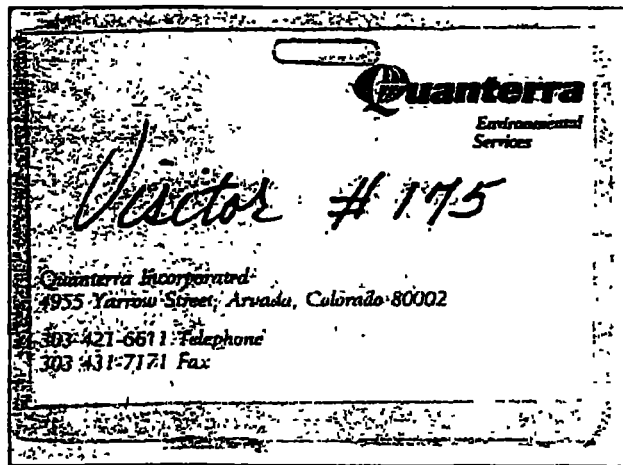
6.2. Appendix II: Example Visitor's Badge

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Appendix I: Example Employee ID Badge



Appendix II: Example Visitor's Badge



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Copy No. _____ **UNCONTROLLED COPY**

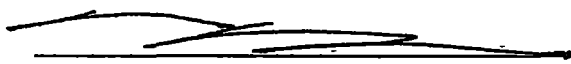
SOP No. DEN-QA-0003
Revision No. 2.0
Revision Date: 4/14/97
Page: 1 of 18

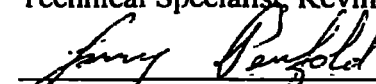
OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

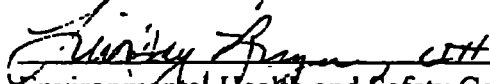
TITLE: SAMPLE RECEIPT AND CHAIN-OF-CUSTODY


(SUPERSEDES: DEN-QA-0003, REVISION 1.0)

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Approved by:  16 - APR - 97
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Approved by:  4/16/97
Laboratory Director, Thomas Daniels

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ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

- 3.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.
- 3.6. Do not open any shipping containers marked "Biohazard" or "Extremely Hazardous" before consulting management personnel.
- 3.7. Shipping containers marked with radiation stickers should be opened under the direct supervision of the Radiation Safety Officer.

4. PROCEDURE

- 4.1. Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.
- 4.2. Routine Sample Receipt
 - 4.2.1. Sample shipments arrive at the central shipping and receiving area. The delivery personnel ring the exterior bell, and Sample Receiving staff unlock the door.
 - 4.2.2. Sign the shipping receipt, and retain a copy of the freight bill for the project file.
 - 4.2.3. Check coolers for radiation as described in "Screening Samples for the Presence of Radioactivity" (SOP#: DEN-CHP-0005).
 - 4.2.4. Move the coolers into the hood before opening them
 - 4.2.5. Use the Sample Checklist (Figure 1) to record these initial checks:
 - 4.2.5.1. Client name and sampling site.
 - 4.2.5.2. Results of radiation check from section 4.2.3 above.
 - 4.2.5.3. Condition of cooler custody seals, if present.
 - 4.2.5.4. Presence of custody form(s).

- 4.2.9.2.3. For non-volatile tests requiring chemical preservation, open the bottle, remove a few tenths of a milliliter of sample with a disposable transfer pipette, touch the pipette tip to wide-range (0-14) pH paper. Discard the pipette and any unused sample.
- 4.2.9.2.4. Record the pH based on the immediate color developed.
- 4.2.9.2.5. Initial the form indicating that the pH check was performed on all samples.
- 4.2.9.2.6. List the sample number and pH for any samples not meeting requirements.
- 4.2.9.3. Number of containers indicated on custody form matches the number received.
- 4.2.9.4. Client sample identifiers on the container labels exactly matches identifiers on the custody form.
- 4.2.9.5. Volatile organic vials are completely filled. no bubbles.
- 4.2.9.6. Proper preservation marked on VOA vials.
- 4.2.9.7. Presence of sediment in samples marked as "filtered" or scheduled for "dissolved" tests.
- 4.2.9.8. Presence of samples scheduled for short holding times (see Figure IV).
- 4.2.9.9. Client supplied extra volume for QC on at least one sample.
- 4.2.9.10. Presence of multiple phases in any of the samples (e.g., solids and liquid or multiple liquid phases). Photograph any multi-phase samples.
- 4.2.10. Verify that the sample volumes meet requirements.
- 4.2.11. Corrective action
 - 4.2.11.1. All discrepancies must be noted on the Sample Checklist.

4.4. Project File and Login Approval

4.4.1. Start a project file with:

- A manila file folder labeled with the project number.
- A photograph of the group of sample containers.
- Chain-of-custody forms.
- Freight bills.
- The completed Sample Checklist.
- A blank Level 3 Checklist, to be used when the final report is assembled.
- Short Hold Forms, if used.
- Printouts of LIMS log-in screens.
- Any paperwork the client supplied with the samples

4.4.2. Deliver the project file to the Project Administrator for review and approval.

4.4.3. The Project Administrator will work with the client and Sample Control, as needed, to resolve any uncertainties in log-in information.

4.4.4. The Project Administrator indicates approval of instructions in LIMS through the "log-release" process.

4.5. Sample Storage

4.5.1. Box the samples by type (i.e., according to the areas within the lab).

4.5.2. With the following exceptions, boxes of samples are stored in the walk-in cooler on the appropriate team shelf.

4.5.2.1. Samples for volatile organics analyses are taken directly to the VOA area refrigerators.

4.5.2.2. Consult the Quarantine Sample SOP for proper storage of samples or coolers labeled with "Quarantine Sample" stickers or other USDA labels.

4.5.2.3. Samples requiring strict internal chain-of-custody are stored in the locked refrigerators.

- 6.4. Figure IV: Short Hold Sheet
- 6.5. Figure V: Walk-In Cooler Sample Sign-out / Sign-in
- 6.6. Figure VI: Internal Chain of Custody
- 6.7. Figure VII: Flow chart
- 6.8. Appendix I: Flow Chart

Figure I - Sample Checklist (Continued)

Sample Checklist	
Project # _____	Duped from Project # _____
Set-up by: _____	Duped Group Codes _____
Logged by: _____	Date: _____
Sample Control Review	
Yes No	Initials
<input type="checkbox"/> <input type="checkbox"/>	1. Chain of custody fill out correctly
<input type="checkbox"/> <input type="checkbox"/>	2. Short holding time worksheet correct
<input type="checkbox"/> <input type="checkbox"/>	3. Sample bottle type correct
<input type="checkbox"/> <input type="checkbox"/>	4. Overflow sample storage in special instructions.
<input type="checkbox"/> <input type="checkbox"/>	5. All login paperwork (sample list, group code report and acceptance letter) is included and correct.
<input type="checkbox"/> <input type="checkbox"/>	6. Trip blanks, equipment blanks, and field blanks have correct aliquot designation.
<input type="checkbox"/> <input type="checkbox"/>	7. Sample description, request list, and acceptance letter in folder.
Comments: Include action taken to resolve discrepancies/problems. Include a hard copy of e-mail or use extra paper if more space is needed. _____	
Initials: _____	
PA or PM Review	
Yes No	Initials Date
<input type="checkbox"/> <input type="checkbox"/>	1. Report input sheet
<input type="checkbox"/> <input type="checkbox"/>	2. Invoice Information
<input type="checkbox"/> <input type="checkbox"/>	3. All discrepancies resolved
<input type="checkbox"/> <input type="checkbox"/>	4. Sample and test matrices correct
<input type="checkbox"/> <input type="checkbox"/>	5. Subcontract paper work correct
<input type="checkbox"/> <input type="checkbox"/>	6. Clear picture of subcontract samples in folder
<input type="checkbox"/> <input type="checkbox"/>	7. Special instructions in LIMS
<input type="checkbox"/> <input type="checkbox"/>	8. Modified component lists checked
<input type="checkbox"/> <input type="checkbox"/>	9. Project due, Turn around time, received and collected date OK.
<input type="checkbox"/> <input type="checkbox"/>	10. Log released.
Comments: _____	
Initials: _____	
Client Contact for Discrepancies	
Person Calling: _____	Date of contact _____
Person Contacted _____	
Client Decision: _____	

Figure III - Guide lines for Sample Bottles and Preservations**GUIDELINES FOR SAMPLE BOTTLES AND PRESERVATIONS***(Unless otherwise noted all samples must be cooled to 4°C/41°F)**Reference: 40 CFR 136.3 Table II*

	Parameter	Container	Preservatives
Aqueous Matrices	1 Alkalinity, BOD, Chloride, Color, Residue, Chlorine, Chromium VI, Conductance, Fluorine, MBAS, Nitrite, Orthophosphate, Total Dissolved/Suspended Solids, Sulfate, Sulfite, pH, Nitrate	1000 ml poly HDPE (NM)	None
	2 Ammonia, COD, TKN, TON, Nitrate + Nitrite, Total Phosphate, TOC, Phenolics	500 ml glass (BR)	2 ml 50% Sulfuric Acid
	3 TPH, Oil & Grease	Two (2) 1000 ml glass (BR)	4 ml 50% Sulfuric Acid
	4 Metals (excluding Hg), Hardness	500 ml HDPE (NM)	10 ml 20% Nitric Acid®
	4 C CLP Metals (excluding Hg), Hardness	1000 ml HDPE (NM)	20 ml 20% Nitric Acid®
	4 M Mercury, Method 7470	500 ml glass (BR)	10 ml 20% Nitric Acid
	5 Gross Alpha, Gross Beta	Two (2) 1000 ml HDPE (NM)	20 ml 20% Nitric Acid®
	6 Total and/or Free Cyanide	250 ml HDPE (NM)	2 ml 50% Sodium Hydroxide
	6 C CLP Total and/or Free Cyanide	500 ml HDPE (NM)	4 ml 50% Sodium Hydroxide
	7 Sulfide	250 ml HDPE (NM)	1 ml 1 N Zinc Acetate and 1 ml 50% Sodium Hydroxide
	9 Volatile Hydrocarbons, GRO	Three (3) 40 ml glass	200 µl Hydrochloric Acid
	11 Purgeable Organics	Three (3) 40 ml glass	200 µl HCl, if sample is chlorinated add 100 µl 2% Sodium Thiosulfate
	12 Base, Neutral, Acid Compounds, Dioxins	Two (2) 1000 ml amber glass (BR)	None
	13 Pesticides, PCBs	Two (2) 1000 ml glass (BR)	None
	14 Herbicides	Two (2) 1000 ml glass (BR)	None
Solid Matrices	15 TOX - Single TOX - Quad	Two (2) 250 ml amber glass Four (4) 250 ml amber glass or one (1) 1000 ml amber glass	1 ml 50% Sulfuric Acid 1 ml 50% Sulfuric Acid or 4 ml 50% Sulfuric acid
	16 Extractable Hydrocarbons, DRO	Two (2) 1000 ml glass (BR)	None
	20, 21 Bulk Water Analysis	1/2 gallon or 1 gallon glass (WM)	None
	30 Organics, TPH, Metals, Radiochemistry, Oil & Grease	500 ml glass (WM)	None
	31 Wet Chemistry, not listed for 30	250 ml glass (WM)	None
	32 Purgeable Organics	125 ml glass (WM)	None
	33 Purgeable Organics (Solid phase)	Two (2) 125 ml glass (WM)	None
	All other analytes (Solid phase)	1000 ml amber glass (WM)	None
	34 Purgeable Organics (Multiphase)	Two (2) 250 ml glass (WM)	None
	All other analytes (Multiphase)	Four (4) 1000 ml amber glass (WM)	None

NM - Narrow Mouth, BR - Boston Round, WM - Wide Mouth HDPE - High Density Polyethylene® - Does not require temperature preservation

Figure IV - Short Hold Sheet

Short Hold Sheet				
Project Number: _____				
Client: _____				
Wet Chemist: _____ <small>signature required</small>				
Time in: _____ Date Received: _____				
<input type="checkbox"/> Raw Data Required <div style="float: right; text-align: right;"> Date Sampled _____ Time Sampled _____ </div>				
Holding Time	Analysis	Method No.	Sample Number(s)	
Immediately	Dissolved Oxygen	360.1		
	Sulfite (SO_3^{2-})	377.1		
	Residual Chlorine	330.1		
	pH†	9040		
	Hydrazine	ASTM D1385		
within 24 Hours	Chromium (VI)	7196		
	Conductivity	120.1		
within 48 Hours	Biological Oxygen Demand	405.1		
	Color	110.2		
	MBAS/Surfactants	425.1		
	Nitrite by Spec†	354.1		
	Orthophosphate by Spec	365.3		
	Nitrate by IC	300.0		
	Orthophosphate by IC	300.0		
	Nitrite by IC	300.0		
	Turbidity	180.1		
	Settleable Solids	SM25401†		
Comments				

† Alkalinity and conductivity may be analyzed simultaneously with pH

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QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION

(SUPERSEDES: REVISION NO. 0 12/11/95)

Prepared by: Quanterra® QA Managers

Approved by:


Director of Quality Assurance

Approved by:


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Approved by:


Vice President Operation Services

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Nonconformance and Corrective Action

SOP No. CORP-QA-0010

Revision No. 1

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1. PURPOSE

- 1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The Quanterra® Quality Assurance Management Plan (QAMP) requires documentation of instances of deviations from established control limits, approved SOPs, or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from plan specifications together with their cause and correction. The NCM may be a written document or a defined electronic formatted document transmitted via e-mail.
- 1.2. This document applies to procedures, services, data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to incoming samples may be documented separately from the NCM process by use of the Condition Upon Receipt (CUR) Form as described in a facility-specific sample receiving/log-in standard operating procedure (SOP). Regardless of which system a lab chooses to use to handle these sample receipt client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.
- 1.3. Identification of suspected adverse conditions (deviations) can be made by laboratory employees in the course of their work or by individuals outside Quanterra's® laboratories through review of records, audit, or proficiency testing.

2. RESPONSIBILITIES

- 2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of Quanterra's® product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.
- 2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors sign NCMs and forward them to a project manager. Together with project managers and Quality Assurance personnel,

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supervisors are responsible for determining the appropriateness of planned corrective actions.

- 2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that reparation and analysis of a sample was done. The project manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.
- 2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for assigning log numbers to the NCMs as received, reviewing all prepared NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.
- 2.5. **Operations/Systems Manager :** The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.
- 2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.
- 2.7. **Corporate QA Director:** The Quanterra® Quality Assurance Director should be notified of any continuing non-conformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

- 3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.
- 3.2. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra® associates.
- 3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra® associate. The situation must be reported immediately to a laboratory supervisor.

4. PROCEDURE

- 4.1. When to Initiate a Nonconformance Memo
 - 4.1.1. Lab associates are to prepare a nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or materials deviate from established specifications. All nonconformances require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).
 - 4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, forms, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs or Quality Assurance Summaries). Any intentional deviation from an SOP must be pre-approved by the QA manager and technical specialist. Any deviation from a SOP or client requirement not previously approved must be documented on an NCM.
 - 4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple project numbers and samples, but normally a separate NCM would be prepared for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be communicated to each project manager. The laboratories may utilize any communication system they feel best satisfies this required communication and maintains the integrity of the NCM process.

4.2. How to Process the NCM Form

- 4.2.1. Enter the information required at the top of the form (see Example Form in Attachment A; printed forms are available from QA). Names or initials must be uniquely identified by using three initials or by spelling out the last name. Use specific test codes (when available) rather than generic codes such as "VOAs" or "Anions." At a minimum enter Project ID, QuantIMS batch number or LIMS QC Lot number if appropriate, in addition to sample numbers. The laboratory generating the NCM is identified as part of the log number (e.g., AUS-97-123). Therefore, separate laboratory location identification is not required on the form.
 - 4.2.1.1. If the NCM is for a holding time violation, the project manager *must* be notified immediately.
 - 4.2.1.2. An instrument tag-out does not require the completion of the NCM if there are no client samples involved (e.g., instrument out of service to perform routine maintenance.) See section 4.2.5.
- 4.2.2. Check appropriate NCM boxes. If box 31—"Other"—is checked, supply a brief description of the problem. Attach further information as necessary.
- 4.2.3. Complete the reverse side of the form concerning root cause and corrective actions. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).
 - 4.2.3.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples. Additional sheets documenting corrective action may be attached to the NCM. Reference the attachment on the NCM.
 - 4.2.3.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.4. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff.

4.2.5. Instrument/Equipment Nonconformance Tag

4.2.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. (See Example in Attachment B.)

4.2.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM and mark box 30. Identify the instrument by name and/or identification number and briefly describe the problem. This information can be recorded on page 2 under "Problem Description/Root Cause". Submit the NCM to the group supervisor.

4.2.5.3. Upon receipt of the NCM, QA assigns the NCM number and provides the number to the analytical group so that the nonconformance tracking number can be recorded in the instrument's maintenance log.

4.2.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

4.2.6. Submit the form to the supervisor responsible for the area indicated at the top of the form.

4.3. Supervisory Review, Approval, and Signature

4.3.1. Review the information provided. Complete or amend the descriptions of root cause and corrective action. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager

needs to be made aware of the issues, the QA staff will notify and document the notification of the operations/systems manager on the form.

- 4.3.2. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired.
- 4.3.3. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the operations/systems managers.
- 4.3.4. The supervisor signs and dates the "Corrective Action Approved By" line and forwards the original form to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager *must* be notified *immediately*.

4.4. Project Manager Review, Client Notification, and Project Documentation

- 4.4.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate on the NCM whether client notification is required or not.
- 4.4.2. Record the result of the client contact or leave blank if client contact was not required. Sign and date the NCM form. This must be done whether client notification is completed or not. The signature documents the project manager as having read and reviewed the NCM.
- 4.4.3. Document any changes to the corrective action plan, and notify the supervisor. The project manager's signature on the front of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.
- 4.4.4. The project manager will forward the original to the QA office within 72 hours for review and closure. The PM may keep a copy for his or her own personal use, but the original must be forwarded to the QA office.
- 4.4.5. If the nonconformance involves analytical work in process, the final report cannot be released until a project manager has signed the NCM.

- 4.4.6. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM (items 25 and 26 on NCM form) and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (LIMS or QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.
- 4.4.7. After the QA staff has closed the NCM, the original of the closed NCM will be filed either in the project file or QA files per laboratory procedure.

4.5. Quality Assurance Review and Tracking

- 4.5.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.
- 4.5.2. NCMs will be reviewed to ensure that the corrective action was completed.
- 4.5.3. A tracking system will also be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:
- NCM log number
 - Date initiated
 - Project number
 - Lab sample ID numbers
 - Method or parameter
 - Nonconformance description and NCM box number
 - Corrective action required
 - Characterization as an anomaly or deficiency
 - Closure of NCM
- 4.5.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic

problems must be documented by the responsible laboratory area along with supporting evidence.

4.5.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.5.6. The signature of the QA manager or designee is required for corrective actions to indicate that the issue has been closed.

4.5.7. The QA office shall maintain a central file/record of all NCMs.

4.6. Externally Generated Corrective Actions

4.6.1. Reports from external quality organizations, such as audit reports, data validation comments, and reports of proficiency tests, shall be considered externally generated. The QA staff is responsible for maintaining the files of externally generated quality reports. If the report is received by an associate other than the QA manager, the report (or copy) must be forwarded to the QA Manager.

4.6.2. Preformatted NCMs are not required to be used to document audit deficiencies or performance evaluation deficiencies. A memorandum format notification by the QA staff and responsible supervisor response with corrective action can be used to document the corrective action process.

4.6.3. The QA manager or designee shall review the reports to identify any deficiencies requiring action.

4.6.4. The QA manager or designee shall work with the responsible supervisor to formulate a corrective action plan. The plan shall be implemented and evidence of corrective action supplied to the QA office.

4.6.5. The QA staff shall verify that the problem has been corrected and prepare a written response to the external organization, if required. Data validation responses shall be coordinated through a project manager for the client.

5. DEFINITIONS

5.1. **Nonconformance:** an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a **deficiency**, or the result of events beyond the control of Quanterra®, then termed an **anomaly**.

A nonconformance exists when:

- 5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a systematic deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required.
- 5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy. *except* in cases where the procedure has been performed according to a client-specified document Quanterra® has agreed to follow (e.g., EPA SOWs and QAPjPs).
- 5.1.3. A practice or procedure is not performed as described according to a client or project document that Quanterra® has agreed to follow.
- 5.1.4. Purchased materials or services are determined to be defective and their use would effect data quality.
- 5.1.5. Holding time violations occur regardless of what or whose actions caused them.
- 5.1.6. A formal NCM is not required for routine instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.
- 5.2. **Corrective action:** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.
 - 5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.
 - 5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Documents

- 6.1.1. Quanterra® Quality Assurance Management Plan (QAMP), current revision:

6.1.2. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

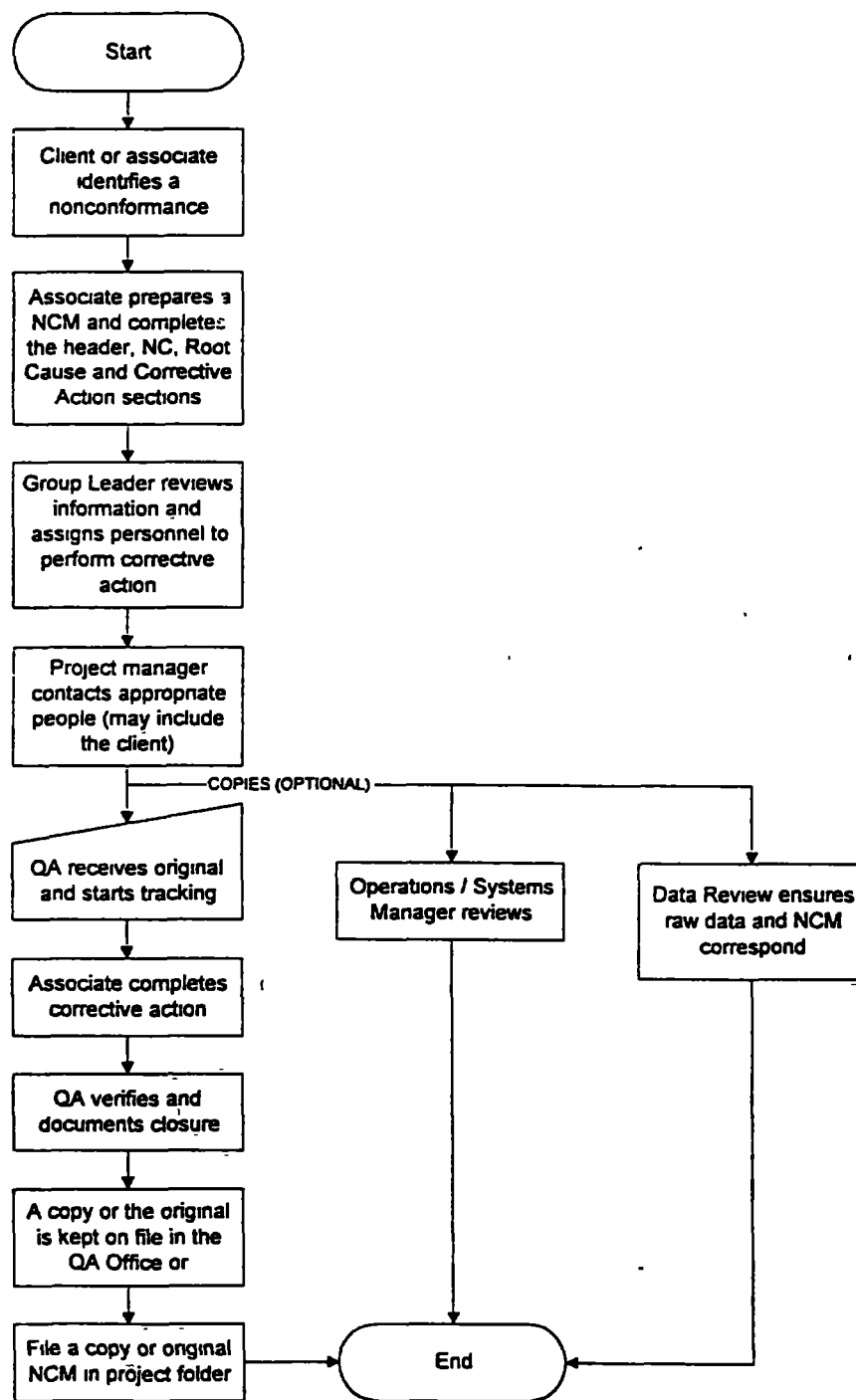
6.1.3. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.2. Appendices

6.2.1. Attachment A: Laboratory Nonconformance Memo (NCM).

6.2.2. Attachment B: Instrument/Equipment Nonconformance Tag Form

6.3. Flow Chart for Internal NCM



UNCONTROLLED COPY

ATTACHMENT A

LOG #: _____

LABORATORY NONCONFORMANCE MEMO (NCM)

Quanterra® Incorporated

Project ID/Client: _____	
NCM Initiated by/Date: _____	Project Manager: _____
Sample Numbers/QC batch or lot numbers: _____	
Tests: _____	
Analytical Area (check appropriate area):	
<input type="checkbox"/> Sample control	<input type="checkbox"/> GC
<input type="checkbox"/> Organic preparation	<input type="checkbox"/> HPLC
<input type="checkbox"/> Inorganic preparation	<input type="checkbox"/> GC/MS
<input type="checkbox"/> Wet chemistry	<input type="checkbox"/> Data review
<input type="checkbox"/> Metals	<input type="checkbox"/> Radiochemistry
<input type="checkbox"/> Reporting	<input type="checkbox"/> _____
Nonconformance (check appropriate area): <i>To be completed by analyst:</i>	
Holding Time Violations (exceeded by _____ days) <i>Category I: Laboratory Independent</i> <input type="checkbox"/> 1. Holding time expired in transit <input type="checkbox"/> 2. Sample rec'd > 48 hrs after sampling, or ½ holding time has expired <input type="checkbox"/> 3. Test added by client after expiration <i>Category II: Laboratory Dependent</i> <input type="checkbox"/> 4. Instrument failure <input type="checkbox"/> 5. Analyst error <input type="checkbox"/> 6. Log-in error <input type="checkbox"/> 7. Miscommunication <input type="checkbox"/> 8. Other (explanation required) _____ <i>Category III: Analysis Reruns (QA/QC)</i> <input type="checkbox"/> 9. Surrogates <input type="checkbox"/> 10. Internal standards <input type="checkbox"/> 11. Spike recoveries <input type="checkbox"/> 12. Blank contamination <i>Category IV: Analysis Reruns (Confirmation)</i> <input type="checkbox"/> 13. Second column <input type="checkbox"/> 14. Contamination check <input type="checkbox"/> 15. Confirmation of matrix effects <input type="checkbox"/> 16. Other (explanation required) _____ <i>Category V: Analysis Reruns (Dilution)</i> <input type="checkbox"/> 17. Over calibration <input type="checkbox"/> 18. Under calibration <input type="checkbox"/> 19. Other (explanation required) _____	Quality Assurance/Quality Control <input type="checkbox"/> 20. QC data reported outside of controls <input type="checkbox"/> 21. Incorrect procedure used <input type="checkbox"/> 22. SOP intentionally modified with QA and tech approval <input type="checkbox"/> 23. Invalid instrument calibration <input type="checkbox"/> 24. Received insufficient sample for proper analysis Incorrect or Incomplete Client Deliverable <input type="checkbox"/> 25. Hardcopy deliverable error <input type="checkbox"/> 26. Electronic deliverable error Reported Detection Limits Elevated Due to: <input type="checkbox"/> 27. Sample matrix: Does not include high analyte content <input type="checkbox"/> 28. Insufficient sample volume <input type="checkbox"/> 29. Other (explanation required) _____ Miscellaneous <input type="checkbox"/> 30. Instrument/equipment Tag-out <input type="checkbox"/> 31. Other (explanation required) _____ _____ _____ _____
Notification (check appropriate area): <input type="checkbox"/> Required <input type="checkbox"/> Not Required <i>To be completed by project manager</i>	
Client notified by (name and date): _____ <input type="checkbox"/> In writing <input type="checkbox"/> By facsimile <input type="checkbox"/> By telephone <input type="checkbox"/> Other (explain) _____	Client's name and response: _____ <input type="checkbox"/> Process "as is" <input type="checkbox"/> Re-sample <input type="checkbox"/> On hold until _____ <input type="checkbox"/> Other (explain) _____
Project manager (signature and date): _____	

Nonconformance and Corrective Action

SOP No. CORP-QA-0010

Revision No. 1

Revision Date: 4/23/97

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QUANTERRA® LABORATORY NONCONFORMANCE MEMO

LOG #:

Corrective Action:		<i>To be completed and reviewed by all associates involved</i>	
Problem Description/Root Cause		Author's initials and date: _____	
Corrective Actions (Short Term)		Author's initials and date: _____	
Corrective Actions to Prevent Reoccurrence (Long Term)			
Corrective Action approved by (Supervisor/Group Leader) and date: _____			
Additional Comments:			
Corrective Action to be completed by (if other than Supervisor/Group Leader): _____			
Date Corrective Action is to be completed: _____			
Quality Assurance Review		<i>To be completed by a QA associate</i>	
<input type="checkbox"/> Anomaly <input type="checkbox"/> Deficiency		<input type="checkbox"/> Notified Ops/Sys Manager (Initials) _____	
<input type="checkbox"/> Further action required: _____			
Further action assigned to: _____			
QA signature: _____		Date: _____	
Corrective Action Verification:		<i>To be completed by a QA associate</i>	
<input type="checkbox"/> Verification not required or requested			
<input type="checkbox"/> Verified / CA completed on: _____ by _____			
<input type="checkbox"/> Cannot verify (specify reason) _____			
Verified by: _____		Date: _____	
Nonconformance Memo Closure:			
QA signature: _____		Date: _____	

The Office of Quality Assurance maintains a copy or the original of this NCM indicating its final status.

ATTACHMENT B

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

<p>QUANTERRA®</p> <p>CAUTION</p> <p>DO NOT USE</p> <p>NONCONFORMING ITEM</p> <p>NCM NUMBER _____</p> <p>AFFECTED ITEM _____</p> <p>_____</p> <p>_____</p> <p>ANALYST _____ DATE _____</p> <p>WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.</p>	
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Control Copy No. UNCONTROLLED COPY

Policy No. QA-012

Revision No. 0

Revision Date: 10/31/96

Page 1 of 7

APPROVAL:


Director, Quality Assurance
Chief Operating OfficerQuanterra® IncorporatedPOLICY NAME

COMPANY WIDE

Technical Data Review
Requirements

Rev. 0, 10/31/96

Supersedes: None

Policy #: QA-012

OBJECTIVE:

To identify the requirements for Level 1 and Level 2 technical data review for organic and metal analyses, thereby ensuring that data released to clients have been correctly produced, processed, and reduced.

SCOPE:

To be enforced and followed throughout the company.

POLICY:

1. All data released to clients or regulators must receive two levels of technical review.
2. The two levels of review must be performed independently and by two different associates.

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for the reviewer to evaluate the overall data quality nor does it remove the responsibility of the reviewer for the data quality.

The review items required to be on the checklist are detailed in the Appendices. Note that these are minimum requirements. Checklists for some methods (e.g., CLP) may have additional elements. In addition, the following items are required for all methods unless an individual element is not part of the method. The checklist must follow the requirements of the SOP used to perform the analysis and the SOP used must be documented on the checklist.

Level 2 Review

In general, the same elements checked for Level 1 review are verified at Level 2 review. Level 2 review may have some additional requirements for manual checking of calculations to ensure that the correct calibration files were used for quantitation.

Note: See Appendices 1-4 for more detail regarding data review according to specific types of tests performed.

RESPONSIBILITIES:

1. The analyst performing data reduction and Level 1 data review bears primary responsibility for the correctness, consistency, and completeness of data released to the client.
2. The associate performing Level 2 review is responsible for ensuring that Level 1 review has been performed fully and correctly.
3. It is the responsibility of the Laboratory Manager to ensure that this policy is implemented for all organic and metal tests.
4. It is the responsibility of the facility QA Manager or designee to perform periodic assessments to ensure that this policy is being followed.

APPENDIX 2

MINIMUM REQUIREMENTS FOR GC/MS DATA REVIEW

Initial Calibration

- Sufficient calibration points used
- BFB/DFTPP meets criteria
- Reason: for removal of any points documented
- %RSD within method limits
- Response factors meet criteria
- Isomeric pairs checked for correct peak assignment
- Any detector saturation
- Automatic integration
- Manual integrations documented

Continuing Calibration

- DFTPP/BFB meets criteria
- % drift of difference meets criteria
- Response factors meet criteria
- Isomeric pairs checked for correct peak assignment
- Correct initial calibration used for quantitation (Level 2 review must verify by recalculation of one or two compounds for the batch)
- Manual integrations documented

Samples

- Any special client requirements followed
- DFTPP/BFB meets criteria
- Samples analyzed within tune time (normally 12 hours from injection of BFB/DFTPP)
- Correct initial calibration used for quantitation (Level 2 review must verify by recalculation of one or two compounds for the batch)
- Any special client requirements reviewed and followed
- Analysis completed within holding time
- Surrogates within QC limits
- Appropriate dilutions performed
- All detected analytes evaluated
- Manual integrations documented
- LCS within QC limits
- Method blank meets QC requirements
- MS/MSD recoveries

Additional Level 2 Requirements

- Recalculate one or two sample results to ensure the correct calibration file(s) were used

APPENDIX 4
MINIMUM REQUIREMENTS FOR METALS DATA REVIEW

Calibration/Run QC

- Initial calibration per SOP
- ICV/CCV meet SOP criteria
- ICB/CCB meet SOP criteria

Sample Results

- Any special client requirements followed
- Analysis within linear range
- Analysis within holding times
- LCS meets criteria
- Appropriate dilution
- Method blank meets criteria
- MS/MSD meets criteria
- Sample duplicate meets criteria
- Serial dilution meets criteria

B

~~SECRET~~

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APPENDIX B
ANALYTICAL STANDARD OPERATING PROCEDURES

INDEX OF STANDARD OPERATING PROCEDURES

SOP NUMBER	SUBJECT	NO. OF PAGES
LM-RMA-3024	Determination of Low Level (Part Per Trillion) PAH and Heterocycles in Water	18
LM-RMA-1112	Total Recoverable Phenolics - City of St. Louis Park	16
CORP-MS-0001DEN	GC/MS Analysis Based on Method 8270B, SW-846	53

STANDARD
OPERATING
PROCEDURE

Subject or Title: DETERMINATION OF LOW LEVEL (PART PER TRILLION)
PAH AND HETEROCYCLES IN WATER Page 1 of 18

SOP No.:
LM-RMA-3024

Revision No.:
7.0

Effective Date:
September 14, 1992

Supersedes: N/A

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1. Summary of the Method

This method has been designed for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion level (ppt,ng/L) in water. The analysis is carried out by isolation of the target analytes by liquid-liquid extraction of the water sample with an organic solvent. Quantitation of the isolated target analytes is performed by gas chromatography mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The compounds listed in Table 1 can be quantitatively determined using this analytical method.

Prepared by:
Phil Tallarico

Date:
September 14, 1992

Management Approval:

Date:
9-14-92

QA Officer Approval:

Date:
9-14-92

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This method has three options for the extraction of the samples depending on the sample type. The three options include two low and one medium level extraction. The low level options have typical reporting limits of 10.0 ppt, with higher surrogate and spike levels in one of the options to accomodate dilutions. The medium level option is eighty times higher in detection limits. A volume of sample dependent of the extraction option chosen is extracted with methylene chloride. Analysis of concentrated extract is performed by gas chromatography/mass spectrometry using the selected ion monitoring scanning mode under electron impact ionization conditions.

2. Interferences

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which contains the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not the specified limits, then interferences may be present.

3. Apparatus and Materials

3.1 Glassware

Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with methanol.

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Glassware should then be solvent rinsed with toluene, acetone and methylene chloride, after extensive rinsing glassware should be air dried, then sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants.

Store glassware inverted or capped with aluminum foil. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

- 3.1.1 Separatory funnel - 2000 and 4000 mL, with Teflon stopcock or continuous liquid liquid extractor, 2000 mL.
- 3.1.2 Drying column - glass funnel with ~10 cm anhydrous sodium sulfate.
- 3.1.3 Concentrator tube, Kuderna-Danish - 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.
- 3.1.4 Snyder column, Kuderna-Danish - Three-ball macro (Kontes K-503000-0121 or equivalent).
- 3.1.5 Evaporative flask, Kuderna-Danish - 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs or clips.
- 3.1.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C. The N-Evap by Organomation Associates, Inc., South Berlin, MA (or equivalent) is suitable.
- 3.1.7 Micro reaction vessels, 2.0 mL (Supelco 3-3295).

3.2 Gas Chromatograph

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

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3.3 Column

A DB-625.5 30 meter fused silica capillary column, or equivalent.

3.4 Mass Spectrometer

A mass spectrometer operating at 70 ev (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

4. Reagents

4.1 Reagent water

Reagent water is defined as water in which the target compounds are not observed at or above the method detection limit.

4.2 Solvents

Acetone, methanol, methylene chloride, cyclohexane - Burdick & Jackson, distilled in glass, or equivalent.

4.3 Sodium sulfate

(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

4.4 Surrogate Spiking Solution

Depending on the extraction option chosen low, low75, or medium a surrogate solution is made by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone and added to the sample prior to extraction with

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methylene chloride. The compounds in the surrogate solutions are naphthalene-d8, fluorene-d10, and chrysene-d12. The low surrogate solution is at 20 ng/mL and 0.5 mL per liter of sample is added. The low75 is at 150 ng/mL and 0.5 mL per liter is added. The medium surrogate solution is at 1000 ng/mL and 1.0 mL is added to the 500 mL sample.

4.5 Internal Standard Solutions

A solution containing ca. 400 ng/mL of each internal standard is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methylene chloride. Fifty microliters of this solution is added to the 0.5 mL extract prior to analysis to give a concentration of the internal standards in the extract of 40 ng/mL.

4.6 Matrix Recovery Standard Spiking Solution

A solution containing the following compounds at the listed concentrations is prepared by weighing an appropriate aliquot of each purified crystal into a volumetric flask and diluting to volume with methanol or acetone. The concentrations of the spiking solution for both the low and medium level extractions are shown below:

<u>Compound</u>	<u>Low Spiking Solution (ng/mL)</u>	<u>Medium Spiking Solution (ng/mL)</u>	<u>Low75 Spiking Solution (ng/mL)</u>
Naphthalene	20	1000	150
Fluorene	20	1000	150
Chrysene	20	1000	150
Indene	20	1000	150
Quinoline	20	1000	150
Benzo(e)pyrene	20	1000	150
2-methylnaphthalene	20	1000	150

The low spiking solution is added at 0.5 mL per liter of sample. The low75 is added at 0.5 mL per 1.0 liter of sample. The medium level spiking solution is added at 1.0 mL per 500 mL of sample.

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5. Sample Preservation, Storage and Holding Times

5.1 Sample Preservation and Storage

The samples must be protected from light and refrigerated at 4°C (+ 2°C) from the time of receipt until extraction and analysis. After analysis, extracts and unused sample volume must be protected from light and refrigerated at 4°C (+ 2°C).

5.2 Holding Times

Samples must be extracted within 5 days of the time of sample receipt. Samples are required to be shipped the same day samples are collected using an overnight carrier.

Extracts must be analyzed within 40 days of extraction.

6. Sample Extraction

6.1 Samples

Samples are extracted at a pH > 12. For the low level extraction, a measured amount of sample, approximately 4 liters, is poured into either two 2-liter continuous liquid-liquid extractors, one 4-liter continuous liquid-liquid extractor, or two 4 liter separatory funnels. The surrogate solution is added and the samples are extracted with methylene chloride. The samples are shaken three times with 80 mL of methylene chloride for the shakeout technique. The samples are allowed to reflux for eighteen hours if the liquid-liquid extractor technique is used for preparation. The extracts from each two-liter fractional extraction (for either technique) are then combined for concentration. The medium level extraction requires that 500 mL of the sample be extracted with methylene chloride for 18 hours in a one liter continuous liquid-liquid extractor or shaken three times with 60 mLs of methylene chloride in a 2-liter separatory funnel. The extracts are passed through an anhydrous sodium sulfate drying column into a 500 mL Kuderna-Danish evaporative concentrator.

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Both low level extracts are concentrated to approximately 0.5 mL and transferred to a 2.0 mL microreaction vessel. The methylene chloride is evaporated using a nitrogen stream. The evaporative concentrator tube is successively rinsed with methylene chloride, the rinsates added to the reaction vessel and the methylene chloride again evaporated. This process is continued until at least five (5) 1 mL rinsings of the tube have occurred.

The final methylene chloride extract for the low level extraction is evaporated to 500 uL. All microreaction vessels are permanently marked at the 500 uL level and additional methylene chloride added, when necessary, to insure a final 500 uL extract volume. The medium level extract is concentrated to 5.0 mL using the same procedure described above. The extract vessels are capped with a Teflon fitted septum cap and stored at 4°C prior to GC/MS analysis.

6.2 Method blank

Method blanks are prepared by treating a 4-L or 500 mL of laboratory reagent water exactly as described above depending on the option chosen. A method blank must be performed once each case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix or whenever samples are extracted by the same procedure, whichever is most frequent.

- * A case is a group or a set of samples collected from a particular site over a given period of time.

6.3 Matrix Recovery Sample

Matrix recovery samples are prepared by spiking a sample as described in section 4.6. The fortified sample is extracted exactly as described above for samples. The laboratory will spike and analyze 5% matrix spike samples (i.e. one matrix spike with every 20 samples).

6.4 Duplicate Sample

For a minimum of 10% of the samples analyzed a duplicate sample will be taken at sampling and a duplicate analysis will be performed. This will be carried out to insure that an estimate of precision will be available.

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7. GC/MS Calibrations

Prior to use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of analysis. Only one level of calibration is used for the two low level and the medium level ppt PAH analyses. The concentrations of standards used to construct the calibration curve are 20, 40, 240, 600, and 1200 ng/mL. The linear range for low level analysis (4 L to 0.5 mL) corresponds to sample concentrations of 2.5, 5, 30, 75, and 300 ng/L. If the concentration of any target compound in a sample exceeds the linear range defined by the above standards, the extracts must be diluted so that the most concentrated analyte falls within the upper half of the calibration curve. The linear range for medium low level analysis (0.5 L to 5.0 mL) corresponds to final sample concentrations of 200, 400, 2400, 6000 and 12000 ng/L. For every 12 hours of GC/MS analysis, the mass spectrometer response for each PAH relative to the internal standard is determined, as described in the Calculations section, using daily check standards at concentrations of 40 ng/mL. Daily response factors for each compound must be compared to the initial calibration curve. If the daily response factors are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

Table 2 contains example RRT data for target compounds.

8. Daily GC/MS Performance Tests

The GC/MS will not be tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. EPA has dropped this requirement for selected ion monitoring (SIM) methods. This allows the laboratory to tune the instrument to maximize the sensitivity for the compounds being analyzed as described below.

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Mass tuning will be performed using the mass calibration compound FC43. Tuning will be performed to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. In the FC43 spectra, the ion abundance of masses 131 and 219 are adjusted to a approximate ratio of 1:1. These two ions are then maximized to be approximately 50 to 70% of the ion abundance of the base mass 69. This procedure maximizes the sensitivity of the instrument in the mass region of interest for the PAH analysis.

9. Gas Chromatography/Mass Spectrometry Analysis

Just prior to analysis an aliquot of internal standard solution is transferred to the sample vial using a 250 uL syringe to give a final internal standard concentration of 40 ng/mL in the extract. Representative aliquots are injected into the capillary column of the gas chromatograph using the following, or similar conditions:

Injector Temp - 250°C
Transfer Line Temp - 290°C
Initial Oven Temp - 30°C
Initial Hold Time - 1 min.
Ramp Rate - 10°C/min.
Final Temperature - 325°C

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each PAH as shown in Table 1. For all compounds detected at a concentration above the MDL, a check is made to insure the confirmation ion is present.

10. Calculations

10.1 Qualitative Identification

Obtain EICPs for the primary m/z and the confirmatory ion. The following criteria must be met to make a qualitative identification:

The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

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For the qualitative identification, the relative retention time (RRT) of unknown peaks fall within +/- 0.06 RRT units.

The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within + 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. In some instances a compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive data includes correct relative retention time and the presents of the secondary ion but the ratio is greater than + 20% of the primary ion which may be caused by an interference of the secondary ion. When the primary ion is not affected by interferences and the decision is agreed to by the reviewer, the compound is flagged with an asterisk (*) on the sample summary sheet.

Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.2 Quantitation

The following formula is used to calculate the response factors of the internal standard to each of the calibration standards.

$$RF = (A_S C_{IS}) / (A_{IS} C_S)$$

where:

- A_S = Area of the characteristic ion for the parameter to be measured.
- A_{IS} = Area of the characteristic ion for the internal standard.
- C_{IS} = Concentration of the internal standard, (ng/mL).
- C_S = Concentration of the parameter to be measured, (ng/mL).

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Based on these response factors, sample extract concentrations for each PAH is calculated using the following formula.

$$C_e = \frac{(A_s)(I_s)}{(A_{is})(RF)}$$

where:

C_e = Sample extract concentration (ng/mL)

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_s = Amount of internal standard added to each extract (ng/mL).

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = (C_e) \times \frac{V_E}{V_S}$$

C = Concentration in Sample (ng/L)

V_E = The final extract volume (mL), and

V_S = The original volume of sample extracted (L).

C_e = The ng/mL amount measured in the analytical extract.

11. Quality Control/Quality Assurance

11.1 GC/MS Tuning

The GC/MS is tuned as described in section 8.0.

11.2 GC/MS Initial Calibration and Continuing Calibration Check

Prior to the use of the method for low level analysis of PAH, a five-point response factor calibration curve must be established showing the linear range of the analysis.

Each calibration standard is analyzed and the area of the primary characteristic ion is tabulated against concentration for each compound. The response factor (RF) for each compound at each concentration level is calculated using the following equation:

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$$RF = \frac{A_s}{A_{is}} \times \frac{C_{is}}{C_s}$$

A_s = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard.

C_{is} = Concentration of the internal standard

C_s = Concentration of the compound to be measured.

For every 12 hours of GC/MS analysis, the mass spectrometer response (RF) for each PAH of interest (Table 1) relative to the internal standard is determined.

These daily response factors for each compound must be compared to the initial calibration curve. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{\overline{RFI} - RFC}{\overline{RFI}} \times 100$$

\overline{RFI} = Average response factor from initial calibration.

RFC = Response factor from current verification check standard.

If the daily response factor are within ± 35 percent of the corresponding calibration curve value the analysis may proceed. If, for any analyte, the daily response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples.

11.3 Method Blank Analysis

A method blank consists of deionized, distilled laboratory water carried through the entire analytical scheme (extraction, concentration, and analysis). The method blank volume must be approximately equal to the sample volumes being processed.

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Method blank analysis are performed at the rate of one per case*, each 14 calendar day period during which samples in a case are received, with every 20 samples of similar concentration and/or sample matrix, or whenever samples are extracted by the same procedure, whichever is most frequent.

If the method blank contains any of the carcinogenic PAHs listed in Table 10-3 at concentrations greater than the method detection limit (MDL), or any other target PAH compound at a concentration 5 times greater than the MDL, the method blank will be considered out of control. Corrective action will include reanalysis of the blank extract, an investigation into laboratory sources of contamination and qualifying that sample data relates to the blank. Blank level contamination should be considered the minimum level of contamination in all samples that are analyzed with the blanks.

* A case is a group or a set of samples collected from a particular site over a given period of time.

11.4 Surrogate Compound Analysis

The laboratory will spike all samples and quality control samples with deuterated PAH surrogate compounds. The surrogate compounds will be spiked into the sample prior to extraction and will measure individual sample matrix effects associated with sample preparation and analysis. Surrogates will include naphthalene-d₈, fluorene-d₁₀, and chrysene-d₁₂.

RMAL will take corrective action whenever the surrogate recovery for any one or more surrogates is outside the following acceptance criteria:

<u>Surrogate</u>	<u>Acceptance Criteria %</u> <u>Low-Level</u>
Naphthalene-d ₈	21-108
Fluorene-d ₁₀	41-162
Chrysene-d ₁₂	10-118

The following corrective action will be taken when required as stated above:

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- a) Check calculations to assure there are no errors;
- b) Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- c) If the surrogate recovery is outside the control limits, the secondary ion may be used to check the quantitation of the surrogate. If the secondary ion meets within the control limits this recovery is reported with flag of # next to the percent recovery.
- d) If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that an interference specific to the surrogate was present that resulted in high recovery and this interference would not affect the quantitation of other target compounds. The presence of this type of interference can be confirmed by evaluating the chromatographic peak shapes in ion intensities of the surrogate.
- e) If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as 0 with the note surrogate diluted out.
- f) Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis of the extract yields surrogate recoveries within the stated limits, then the reanalysis data will be used. Both the original and reanalysis data will be reported.

11.5 Matrix Spike Analysis

The laboratory will spike and analyze 5% matrix spike samples. RMAL will spike seven representative compounds into water. These compounds and the spiking levels as listed in section 4.5. The initial matrix spike criteria for data validity are as follows:

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SPIKE COMPONENT	ACCEPTANCE CRITERIA
1H-INDENE	20-150
NAPHTHALENE	20-150
QUINOLINE	20-150
2-METHYLNAPHTHALENE	20-150
FLUORENE	69-118
CHRYSENE	20-132
BENZO(E)PYRENE	20-150

One compound is allowed to be below the above acceptance criteria. The average recovery for the spike pair must also fall into the above criteria with one compound being allowed below the acceptance criteria.

Criteria for data validity for each individual matrix spike compound will be developed as data is collected and will be updated annually.

If the matrix spike criteria are not met, the matrix spike analysis will be repeated. If the subsequent matrix spike analysis meets the criteria, then the reanalysis data will be used. If not, the data for the sample will be reported but qualified as being outside the acceptance criteria of the method. Both the original and reanalysis data will be reported.

11.6 Duplicates

The laboratory will analyze 10% duplicate samples. Percent difference between duplicates will be calculated for each detected compound. Corrective action will be performed if the relative difference is greater than 70% for target compounds.

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COMPOUNDS AND MS QUANTITATION MASS IONS*

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion</u>	<u>Internal Standard Reference</u>
<u>Polynuclear Aromatic Hydrocarbons (PAH)</u>			
Naphthalene	128	102	1
Acenaphthylene	152	151	1
Acenaphthene	154	153	1
Fluorene	166	165	1
Phenanthrene	178	176	2
Anthracene	178	176	2
Fluoranthene	202	200	2
Pyrene	202	200	2
Benzo(a)anthracene	228	226	3
Chrysene	228	226	3
Benzofluoranthenes	252	250	3
Benzo(a)pyrene	252	250	3
Indeno(1,2,3,cd)pyrene	276	274	3
Dibenz(a,h)anthracene	278	279	3
Benzo(g,h,i)perylene	276	274	3

Internal Standards

1) Acenaphthene-d10	164	--
2) Phenanthrene-d10	188	--
3) Benzo(a)pyrene-d12	264	--

* The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICP's must fall within +/- 20% of the relative intensities of these masses in a reference mass spectrum.

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TABLE 1 (Continued)

Surrogates

1) Naphthalene-d8	136	134	1
2) Fluorene-d10	176	174	1
3) Chrysene-d12	240	236	3

<u>Compound</u>	<u>Quantitation Mass Ion</u>	<u>Confirmation Ion</u>	<u>Internal Standard Reference</u>
<u>Heterocycles and Other PAH</u>			
Indene	116	115	1
Indole	117	90	1
2,3-dihydroindene	117	118	1
2,3-benzofuran	118	90	1
Quinoline	129	102	1
Benzo(b)thiophene	134	89	1
2-methylnaphthalene	141	115	1
1-methylnaphthalene	141	115	1
Biphenyl	154	153	1
Carbazole	167	166	2
Dibenzofuran	168	139	1
Acridine	179	178	2
Dibenzothiophene	184	139	2
Perylene	252	250	3
Benzo(e)pyrene	252	250	3
7,12-Dimethylbenz(a)anthracene	256	241	3
3-Methylcholanthrene	268	252	3

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TABLE 2

RELATIVE RETENTION TIMES AND CONFIDENCE FOR THE COMPOUNDS
ASSOCIATED WITH THE LOW LEVEL PAH AND HETEROCYCLE METHODOLOGY

	Absolute Retention Time (minutes)	Avg. RRT	SD	% RSD	95% Confidence Limits
Benzofuran	8:03	0.550	0.015	2.807	0.520-0.580
Dihydroindene	8:45	0.590	0.016	2.765	0.558-0.622
Indene	8:54	0.598	0.016	2.699	0.566-0.630
Naphthalene-d8 (Surr.)	11:14	0.733	0.017	2.289	0.699-0.767
Naphthalene	11:16	0.735	0.017	2.289	0.701-0.769
Benzo(b)thiophene	11:25	0.743	0.017	2.258	0.709-0.777
Quinoline	12:06	0.783	0.017	2.140	0.749-0.817
Indole	12:55	0.824	0.018	2.167	0.788-0.860
2-methylnaphthalene	12:59	0.832	0.017	2.084	0.798-0.866
1-methylnaphthalene	13:15	0.848	0.017	2.055	0.814-0.882
Biphenyl	14:12	0.901	0.017	1.921	0.867-0.935
Acenaphthylene	15:15	0.962	0.018	1.822	0.927-0.988
Acenaphthene	15:44	0.988	0.018	1.849	0.952-1.024
Dibenzofuran	16:09	1.011	0.018	1.791	0.975-1.047
Fluorene-d10 (Surr.)	16:57	0.872	0.015	1.735	0.842-0.902
Fluorene	17:01	0.875	0.015	1.745	0.845-0.905
Dibenzothiophene	19:08	0.974	0.016	1.617	0.942-1.006
Phenanthrene	19:28	0.988	0.016	1.589	0.956-1.020
Anthracene	19:34	0.994	0.016	1.597	0.962-1.026
Acridine	19:42	0.999	0.016	1.572	0.967-1.031
Carbazole	20:02	1.013	0.015	1.487	0.983-1.043
Fluoranthene	22:32	1.130	0.017	1.461	1.096-1.164
Pyrene	23:07	1.157	0.017	1.443	1.123-1.191
Benz(a)anthracene	26:16	0.873	0.012	1.325	0.849-0.897
Chrysene-d12 (Surr.)	26:18	0.874	0.012	1.320	0.850-0.898
Chrysene	26:22	0.876	0.012	1.320	0.852-0.900
Benzo(a)fluoranthene	29:00	0.960	0.014	1.501	0.932-0.988
Benzo(e)pyrene	29:34	0.984	0.016	1.590	0.952-1.016
Benzo(a)pyrene	29:44	0.988	0.016	1.615	0.956-1.020
Perylene	29:55	0.996	0.016	1.644	0.964-1.028
Indeno(1,2,3 cd)pyrene	32:31	1.114	0.025	2.276	1.064-1.164
Dibenz(ah)anthracene	32:36	1.113	0.031	2.743	1.051-1.175
Benzo(ghi)perylene	33:17	1.149	0.028	2.422	1.093-1.205

STANDARD
OPERATING
PROCEDURE

Subject or Title: Recoverable Phenolics - City of St. Louis Park Page 1 of 16
(Manual)

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LM-RMA-1112

Revision No.:
1.0

Effective Date:
June 13, 1990

Supersedes: Original

1. Scope and Application

- 1.1 This method measures steam-distillable phenolic materials which react with the color reagents under the conditions of the analysis.
- 1.2 The detection limit is 5 ug/L as Phenol.
- 1.3 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.
- 1.4 The range extends to 0.1 mg/L. The range can be extended by dilution of the samples.
- 1.5 Approximate preparation time is 2 hours for a group of 10 samples. Analytical time is about 15 minutes per sample.

2. Summary of Method

The sample is acidified and distilled to separate phenolics from interfering compounds. Phenolics in the distillate react with 4-aminoantipyrine in the presence of potassium ferricyanide at pH 10 to form a reddish-brown dye, which is extracted into chloroform and measured colorimetrically at 460 nm.

3. Comments

3.1 Interferences

- 3.1.1 Most direct interferences are eliminated by distillation of an acidified sample. Phenolic compounds distill with the water but interfering compounds do not.

Prepared by:
Lindsay Breyer

Date:
June 13, 1990

Management Approval:

Date:

QA Officer Approval:

Date:

Anne Lano

John Zimmerman

June 14, 1990

June 15, 1990

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- 3.1.2 Some phenolic compounds are not steam-distillable and will not be determined.
- 3.1.3 The colors produced by various phenolic compounds are not the same, so the response will depend on the compounds actually present in the samples. Phenol has been selected as the calibration standard since it is not possible to reproduce the mixture of compounds present in the sample. The result obtained will represent the minimum concentration of phenolics present in the sample.
- 3.1.4 Interference from sulfur compounds is eliminated by acidification and addition of copper sulfate.
- 3.1.5 Oxidizing agents such as chlorine will oxidize phenolic compounds and must be removed.
- 3.1.6 Oil may distill over and interfere with the analysis.
- 3.1.7 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

4. Safety Issues

- 4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.
- 4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.
- 4.3 Wear gloves, apron, and face shield when performing distillations. Distillations are to be performed under the slot hood.
- 4.4 Phenol is extremely toxic and can be absorbed through the skin. Handle only in a fume hood and wear gloves. In case of skin contact, flush with water for at least 15 minutes. Notify your supervisor or safety officer of any exposures.

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- 4.5 Extractions are to be performed in a fume hood. Wear gloves and apron. Take care to keep chloroform vapors confined to the hood.
- 4.6 Samples, reagents and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

5. Samples Collection and Preservation

- 5.1 Samples are to be collected in glass containers and preserved by adding sulfuric acid to pH < 2 and refrigerating at 4°C.
- 5.2 The holding time is 28 days.

6. Apparatus

- 6.1 All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller, and associated adapters and hardware.
- 6.2 Recirculating chiller.
- 6.3 pH meter and electrode.
- 6.4 Separatory funnels, 500 mL, with supporting rack.
- 6.5 Porcelain spot-test plate.
- 6.6 Spectrophotometer with 2 cm cells.
- 6.7 Filter funnels.
- 6.8 Filter paper, Whatman 41.
- 6.9 Micropipettes with disposable tips, 10 μ L, 20 μ L, 1 mL.
- 6.10 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Sulfuric Acid, 50%

Slowly add 500 mL concentrated sulfuric acid to 500 mL deionized water with constant mixing and cool. The reaction is very exothermic and should be done with extreme caution.

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7.2 Boiling stones.

7.3 Copper Sulfate, 10%

Dissolve 100 g cupric sulfate 5-hydrate in deionized water and dilute to 1000 mL.

7.4 Ferrous Ammonium Sulfate Solution

Add 1 mL concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.5 Buffer Solution

Dissolve 16.9 g ammonium chloride in 143 mL concentrated ammonium hydroxide and dilute to 250 mL with deionized water. Prepare this solution in a hood. Two milliliters of this solution should adjust the pH of 100 mL distillate to 10.

7.6 Aminoantipyrene Solution

Dissolve 2.0 g of 4-aminoantipyrene in deionized water and dilute to 100 mL.

7.7 Potassium Ferricyanide Solution

Dissolve 8 g potassium ferricyanide in deionized water and dilute to 100 mL.

7.8 Phenol Stock Standard, 1000 mg/L

Dissolve 1.000 g phenol in deionized water and dilute to 1000 mL.

7.9 Phenol Intermediate Standard, 1.0 mg/L

Dilute 1.0 mL 1000 mg/L Stock Standard to 1000 mL with deionized water.

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7.10 Working Standards

Dilute the 1.0 mg/L Intermediate Standard with deionized water as follows:

Aliquot (mL)	Final Vol. (mL)	Conc. (mg/L)
0	200	Blank
1.0	200	0.005
2.0	200	0.010
4.0	200	0.020
10.0	200	0.050
20.0	200	0.100

Note: The standards are not distilled with the samples.

7.11 pH test strips

7.12 Starch/iodide test strips

7.13 Lead Acetate test strips

8. Procedure

8.1 Sample Preparation

- 8.1.1 Measure and record the pH of all water samples. pH test strips may be used.
- 8.1.2 Check for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet.
- 8.1.3 Check for sulfide using lead acetate test strips. A dark color indicates the presence of sulfide. Record the result on the bench sheet.
- 8.1.4 Measure 200 mL sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 2.0 g and add 200 mL deionized water. Be sure to adjust the pH of soil and waste samples before distillation. Record the exact weight on the bench sheet.
- 8.1.5 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

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- 8.1.6 If the pH is not < 2 , add 50% sulfuric acid dropwise until it is.
- 8.1.7 If the sulfide test was positive, add 2 mL 10% copper sulfate.
- 8.1.8 Assemble the distillation apparatus, turn on the cooling water and hood, and start the distillation. Capture the distillate in a 250 mL beaker.
- 8.1.9 When 150 to 175 mL distillate has been collected, turn off the heating mantle and allow to cool.
- 8.1.10 Add 25 to 30 mL deionized water and resume distillation until 200 mL has been collected. Turn off the heating mantle and clean out the flask when cool. Do not over distill the samples as this will lead to interferences in the analysis.
- 8.1.11 Transfer the distillates to 250 mL glass bottles with teflon caps and refrigerate until they are analyzed.

8.2 Spot Test

- 8.2.1 Place 1 mL aliquots of each sample in the wells of a porcelain spot test plate. Also run a blank (deionized water) and the 0.10 mg/L standard.
- 8.2.2 Add 20 μ L buffer solution to each well and stir.
- 8.2.3 Add 10 μ L aminoantipyrene solution and stir.
- 8.2.4 Add 10 μ L potassium ferricyanide solution and stir.
- 8.2.5 Compare the color of the samples to the color of the blank and standard. Any samples appearing darker than the standard will require dilution prior to analysis. Make note of these on the bench sheet along with the estimated dilution required. If necessary, dilute the sample and spot check the dilution.

8.3 Dilution Technique

- 8.3.1 Since the sample volumes may not be exactly 200 mL after distillation, it is not possible to make dilutions volumetrically. Dilutions must be done on a weight basis.

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- 8.3.2 Place a beaker on a top loading balance and zero it.
- 8.3.3 Pour the entire sample into the beaker and note the weight.
- 8.3.4 Divide the weight by the required dilution factor to determine the sample weight to be analyzed. For example, if there are 205 g distillate and a 10x dilution is needed, 20.5 g of the distillate should be analyzed.
- 8.3.5 Measure out this weight of sample for analysis and dilute to a total volume of 200 mL. Return the unused portion of the sample to the original container. Record all dilutions made on the bench sheet.

8.4 Analysis of Samples

- 8.4.1 Place 200 mL sample (or standard) in a 500 mL separatory funnel. Analysis should be performed in a hood.
- 8.4.2 Add 4 mL buffer solution and mix.
- 8.4.3 Check the pH with a pH meter (pH paper is not sensitive enough). The pH should be 10 ± 0.2 . If necessary, adjust the pH by dropwise addition of ammonium hydroxide or hydrochloric acid.
- 8.4.4 Add 2 mL aminoantipyrene solution and mix.
- 8.4.5 Add 2 mL potassium ferricyanide and mix.
- 8.4.6 Wait 3 minutes, then add 25 mL chloroform.
- 8.4.7 Shake the separatory funnel 10 times. Vent chloroform fumes into the hood. Then allow the phases to separate.
- 8.4.8 Shake the funnel another 10 times and let the chloroform settle.
- 8.4.9 Filter the chloroform extracts through filter paper into 2 cm cuvettes.
- 8.4.10 Measure and record the absorbances at 460 nm, zeroing on chloroform; not the blank.

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- 8.4.11 If any samples measure higher than the highest standard, up to a 5x dilution may be made on the extract with chloroform. Record the dilution on the bench sheet and make it clear that the chloroform extract was diluted, as the calculation will be affected.

9. QA/QC Requirements

9.1 QC Samples

- 9.1.1 A blank (deionized water) is required with every batch of 20 less samples. The blank must be taken through the entire prep and analysis with the samples. Additional blanks, termed "Initial Calibration blank" (ICB) and "Continuing Calibration Blank" (CCB) are also analyzed. These blanks are used only to evaluate the determinative step and are not distilled. They are analyzed at a frequency of one ICB per 20 samples and one CCB per 10 samples.

- 9.1.2 The calibration is verified by the analysis of two different laboratory check standards. An "Initial Calibration Verification" (ICV) check standard is analyzed at a frequency of one per 20 samples. This check is carried through the entire procedure, including the distillation step. The measured value from this check standard must be between 75% and 125% of the true value.

A "Continuing Calibration Verification" (CCV) check standard is analyzed at a frequency of one per 10 samples. This standard is used to verify the determinative step only. The measured value must be between 85% and 115% of the true value.

If the measured values from the check standards are not within control limits, the system is out of control and corrective action must be performed.

- 9.1.3 Save the original blank and standards; new ones do not have to be extracted.
- 9.1.4 Duplicate analyses are performed at a frequency of 5%. Corrective action is performed if the relative difference from the duplicate analysis is greater than 70%.

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- 9.1.5 Matrix spikes will be performed at a frequency of 5%. The spike level is 50 ug/L. The recovery of the matrix spike must be between 75% and 125%. Corrective action is performed if these criteria are not achieved.

9.2 Acceptance Criteria

- 9.2.1 An acceptable blank must not contain phenolics above the nominal reporting limit of 5 ug/L. If any of the blanks contain phenolics above 5 ug/L, the system is out of control and corrective action must be performed.
- 9.2.2 ICV and CCV recoveries must be 75 - 125%.
- 9.2.3 Matrix spike recoveries must be between 75% and 125%.
- 9.2.4 The calibration curve must have a correlation coefficient of at least 0.995.

9.3 Corrective Action Required

- 9.3.1 The color reaction is very sensitive to pH and the extraction technique. Check the pH of all samples before developing the color. Use the same extraction technique for all samples and standards.

10. Calculations

- 10.1 Subtract the blank absorbance from the standard and sample absorbances. If the chloroform extract was diluted, divide the blank absorbance by the dilution factor before subtracting.
- 10.2 Enter the corrected standard readings into a linear least squares program to determine the calibration curve.
- 10.3 Calculate the sample results from their corrected absorbances using the least squares program. Multiply by any dilutions made during prep or analysis.

11. Data Reporting Deliverables

The data packages for total phenolics shall as closely follow CLP deliverables for inorganic analysis as possible. Reports shall contain all applicable CLP forms as well as the associated raw analytical data. The package includes Forms I - III, V and VI (results, initial and continuing calibration verification, blanks, matrix spike and duplicate). Examples of these forms are included in this SOP (Pages 11-16). The report shall be organized as described in CLP SOW 7/88.

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12. References

12.1 Method source: EPA Methods 420.1, 420.2

12.2 Deviations from source method and rationale

- 12.2.1 There is a discrepancy between the preservation methods and holding times given in the method and those given in the table of containers and preservatives at the front the methods book. We have chosen to use sulfuric acid to adjust the sample pH to 2 since this has been done traditionally at RMAL.
- 12.2.2 The size of the distillation apparatus and volumes of sample and reagent were reduced to conserve space and speed up the analysis.
- 12.2.3 Provisions have been made for dilution of the chloroform extracts up to 5x. This is sometimes necessary for samples which are overrange and cannot be reprepmed due to limited sample volume or other reasons.

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June 13, 1990COVER PAGE - INORGANIC ANALYSES DATA PACKAGELab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \ Project: \Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \SOW No. 7/88

RMA Sample No.

Client Sample ID.

///

///

PARAMETERSMETHODDETECTION LIMITSOURCE

Phenolics

420.1

5 ug/L

1

COMMENTS:

\

SOURCE:

1="Methods for Chemical Analysis of Water and Wastes," USEPA-EMSL, Cincinnati.

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____ Name: _____

Date: _____ Title: _____

SOP No.:
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June 13, 1990I
INORGANIC ANALYSIS DATA SHEETLab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \Lab Code: ENSECO Case No.: \ SAS No.: \

Matrix (soil/water): \ SDG No.: \

Level (low/medium): \

% Solids: \

Project: \

Date Received: \

Concentration Units (ug/L or mg/kg dry weight): \

RMA Sample No. \ Client Sample ID. \

Analyte	Concentration	C	Q	M
Phenolics				

Color Before: \

Clarity Before: \

Texture: \

Color After: \

Clarity After: \

Artifacts: \

Comments:

\
\

SOP No.:
LM-RMA-1112



Revision No.:
1.0

Effective Date:
June 13, 1990

2A
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: ROCKY MOUNTAIN ANALYTICAL

Contract: \

Lab Code: ENSECO

Case No.: \

SAS No. \

SDG No.: \

Initial Calibration Source: \

Continuing Calibration Source: \

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					M
	True	Found	%R(1)	True	Found	%R (1)	Found	%R (1)	
Phenolics									

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1.0

Effective Date:
June 13, 1990

3
BLANKS

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Preparation Blank Matrix (soil/water): \

Preparation Blank Concentration Units (ug/L or mg/kg): \

Analyte	Initial Calib. Blank (ug/L)		Continuing Calibration Blank (ug/L)						Preparation Blank		
		C	1	C	2	C	3	C		C	M
Phenolics											

SOP No.:
LM-RMA-1112Revision No.:
1.0Effective Date:
June 13, 19905A
SPIKE SAMPLE RECOVERYRMA Sample No.
Client Sample ID.Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Matrix: \ Level (low/medium): \

% Solids for Sample: \

Concentration Units (ug/L or mg/kg dry weight): \

Analyte	Control Limit % R	Spiked Sample Result (SSA) C		Sample Result (SA) C		Spike Added (SA)	% R	Q	M
Phenolics									

Comments: \

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LM-RMA-1112



Revision No.:
1.0

Effective Date:
June 13, 1990

6
DUPLICATES

RMA Sample No.
Client Sample ID.

Lab Name: ROCKY MOUNTAIN ANALYTICAL Contract: \

Lab Code: ENSECO Case No.: \ SAS No.: \ SDG No.: \

Matrix (soil/water): \ Level (low/medium): \

% Solids for Sample: \ % Solids for Duplicate: \

Concentration Units (ug/L or mg/kg dry weight): \

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Phenolics								

Controlled Copy
Copy No: UNCONTROLLED COPY

SOP No: CORP-MS-0001DEN
Revision No: 1.1
Revision Date: 10/25/96
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LABORATORY-SPECIFIC


QUANTERRA DENVER STANDARD OPERATING PROCEDURE


TITLE: GC/MS ANALYSIS BASED ON METHOD 8270B, SW-846

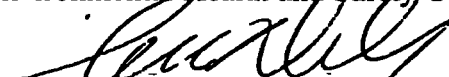
(SUPERSEDES: Revision 1)


Prepared by: Richard Burrows

Reviewed by:  10-28-96
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Approved by:  10/25/96
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Approved by:  10/29/96
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Approved by:  10/29/96
Corporate Technology and/or Corporate Quality Assurance

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TABLE 6	DFTPP Ion Abundance Criteria
TABLE 7	Characteristic Ions, Primary Standard
TABLE 8	Characteristic Ions, Appendix IX Standard
TABLE 9	8270B LCS Compounds
TABLE 10	TCLP LCS Compounds
TABLE 11	8270B Surrogate Compounds
TABLE 12	Calibration Levels, Primary Standard
TABLE 13	Calibration Levels, Appendix IX Standard
TABLE 14	Initial Demonstration Accuracy and Precision Limits

2. SUMMARY OF METHOD

- 2.1. A measured amount of sample is serially extracted with methylene chloride using a separatory funnel, a continuous extractor, Accelerated One-Step™, or with methylene chloride / acetone using Soxhlet or sonication. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3. DEFINITIONS

- 3.1. CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.
- 3.2. SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.
- 3.3. Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.
- 3.4. Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.
- 3.5. LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

- 5.1.1. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 5.1.2. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:
 - 5.1.2.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.
- 5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.
- 5.1.4. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported immediately to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

- 6.1.1. Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.
- 6.1.2. Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5- μ m film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.
- 6.1.3. Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for

- 7.2.1. Internal Standards are added to all standards and extracts at 40 µg per mL of extract. For example, if the volume of an extract used was 200 µL, 20 µL of the 400 µg/mL internal standard solution would be added.
- 7.3. Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.
- 7.4. GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50 µg/mL.
- 7.5. Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.
- 7.6. Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.
- 7.7. The working standards listed in 7.1 to 7.6 should be refrigerated at $4 \pm 2^{\circ}\text{C}$ when not in use. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $4 \pm 2^{\circ}\text{C}$. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature.

8. SAMPLE PRESERVATION AND STORAGE

- 8.1. Reference appropriate facility SOP for sample bottle preservation and storage.
- 8.2. Samples are stored at $4 \pm 2^{\circ}\text{C}$. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)
- 8.3. Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

repreparation and reanalysis of all samples in the QC batch. The following special situations, while requiring documentation, may allow qualified data to be reported without reanalysis:

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.
- If analyte concentration in samples is greater than 20 times MB concentration, the data may be reported with qualifiers after consultation with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

- 9.3.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.
- 9.3.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.
- 9.3.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.
- 9.3.4. Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4. Instrument Blank

- 9.4.1. Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

must be taken. Corrective action will normally include reparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Surrogates

9.7.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at either 100 or 150 ng on-column, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2. If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.7.3. If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

10.3. Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1. Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2. The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be $< 20\%$. Refer to section 12 for the appropriate calculations.

10.4. Initial Calibration

10.4.1. Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2. Compounds should be assigned to the IS with the closest retention time.

10.4.3. Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Add the internal standard mixture at 40 μg per mL of calibration standard. (For example, if the volume of the calibration standard used is 1 mL, add 100 μL of the 400 $\mu\text{g/mL}$ internal standard solution). The working range of the GC/MS system is defined by calibration standards at concentrations of 20 to 160 $\mu\text{g/mL}$. The exact concentrations of all analytes are listed in tables 12 and 13.

10.4.4. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. No sample analysis may be performed unless these criteria are met.

- 10.4.7. If the %RSD of all the analytes in the calibration is $\leq 15\%$, then all analytes may use average response factor for calibration. If an analyte has a %RSD $> 15\%$, a calibration curve will be used instead of average response factor.

10.4.8. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Alternately, a linear fit forced through zero may be used if this improves accuracy at the low end of the curve.

- 10.4.9. If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10. Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

10.5. Continuing Calibration

- 10.5.1. At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

- 10.5.2. Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. The level 3 calibration standard is used for the continuing calibration.

- 10.5.3. The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05 .
- The percent drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.

- 11.2.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution.

11.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

- 11.4. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at $4 \pm 2^{\circ}\text{C}$, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5. Internal Standard Criteria for Samples

Internal standard response in each sample should be within 50% to 200% of the response in the preceding continuing calibration standard.

- 11.5.1. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis may not be necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. In any event, the reason for accepting the sample analysis must be documented. Some clients may require reanalysis of all samples with internal standard criteria outside the 50-200% criteria. Consideration should be given to reanalyzing at a dilution to reduce matrix effects. It is only necessary to reanalyze once to confirm matrix effect.

11.9.2. Major Maintenance

11.9.2.1. If minor maintenance does not result in acceptable chromatography, it may be necessary to change the column.

11.9.2.2. Clean ion volume or repeller. Cleaning is indicated when DFTPP does not pass spectral criteria.

11.9.2.3. Clean source. If after cleaning ion volume or repeller, tune still does not meet criteria, or if overall sensitivity is poor, the source is removed and cleaned.

11.9.2.4. Replace filaments, replace filters and change pump oil. All of these may be done at the same time as source cleaning. The frequency of changing filters and pump oil is about every 6-12 months.

11.9.2.5. A multiplier gain check is performed if sensitivity is still poor and/or analyst suspects that the multiplier is going bad.

11.9.2.6. Mass calibration is performed if the analyst notices mass misassignments.

11.9.2.7. Refer to the manufacturer's manual for specific guidance.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

- The relative intensities of the major ions should agree within $\pm 20\%$. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)
 - Molecular ions present in the reference spectrum should be present in the sample spectrum.
 - Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
 - Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
 - Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.
- 12.4. Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine

12.5.3. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1. Linear fit

$$C_{ex} = A + B \frac{(R_x C_n)}{R_n}$$

Where: C_{ex} = Concentration in extract, $\mu\text{g/mL}$

R_x = Response for analyte

R_{is} = Response for internal standard

C_{is} = Concentration of internal standard

A = Intercept

B = Slope

12.5.3.2. Quadratic fit

$$C_{ex} = A + B \left(\frac{R_x C_n}{R_n} \right) + C \left(\frac{R_x C_n}{R_n} \right)^2$$

Where: C = Curvature

12.5.3.3. Average response factor

Alternatively, if the %RSD of the response factors of the analytes in the initial calibration is $\leq 15\%$, the average response factor from the initial calibration may be used.

$$C_{ex} = \frac{R_x C_n}{\overline{R_n RF}}$$

Where: \overline{RF} = Average response factor

12.7. Relative % Difference calculation for the MS/MSD

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12.8. Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_x = Concentration of the compound being measured ($\mu\text{g/L}$)

C_{is} = Concentration of the specific internal standard ($\mu\text{g/L}$)

12.9. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

$RF=1$

12.10. Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDE area} + \text{DDD area}}{\text{DDT area} + \text{DDE area} + \text{DDD area}} \times 100$$

The total ion current areas are used for this calculation

- 13.5. Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14. POLLUTION PREVENTION

This section is not applicable to this procedure.

15. WASTE MANAGEMENT

Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

- 16.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270B.
- 16.2. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Spike concentrations and Injection Volume

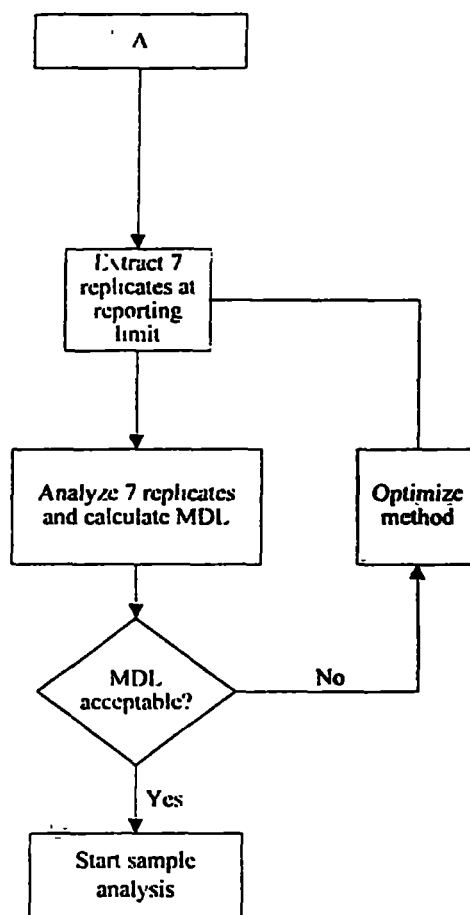
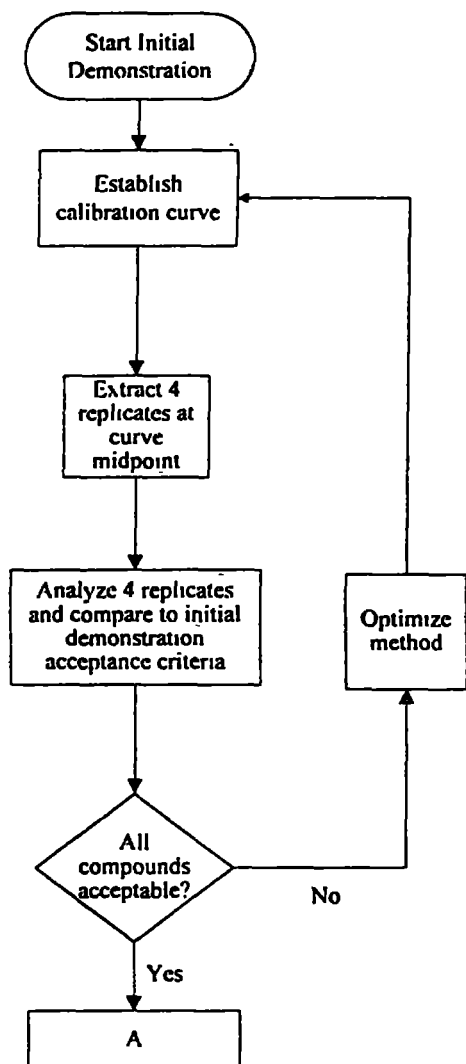
Some of the recommended spike concentrations in the reference method have been changed to bring them within the calibration range of the instrument.

- 17.1.2. The concentration of the internal standard spiking solution is more dilute than that recommended in the reference method. This eliminates the risk of precipitation of the internal standards from solution when the standard is stored in the freezer, and also allows more accurate internal standard addition to small volumes of extract.

- 17.1.3. The internal standard control criteria of 50% to 200% is applied to each sample, rather than the subsequent continuing calibration standard as listed in the reference method. This is a more rigorous criterion than that in the reference method.

17.5. Flow Diagrams

17.5.1. Initial demonstration and MDL



17.6. Tables

Table 1

Quanterra Primary Standard¹ and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane) ²	108-60-1	10	330
4-Methylphenol ³	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600

Table 2

Quanterra Appendix IX¹ Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate ²	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin ²	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp ²	3689-24-5	50	1600
Phorate ²	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate ³	2303-16-4	20	660
Dimethoate ²	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton ²	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion ²	298-00-0	50	1600

Table 2R

Quanterra Refinery List¹ Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Quinoline	91225	10	330
Benzenthrol	108985	100	3300
1-Methylnaphthalene	90120	10	330
1H-Indene	95136	10	330
Dibenz(a,h)acridine	226368	10	330
6-Methylchrysene	170857	10	330
* added compounds for refinery list			

¹ The Refinery standard contains additional analytes required for the Refinery list. The primary standard and Appendix IX standard must also be analyzed to include all the Appendix IX list.

Note: Some of these compounds may be included in the primary standard.

Table 3

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Analyte	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene ¹	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X

¹ 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for Quanterra Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5				X
Parathion ²	56-38-2				X
Isodrin ¹	465-73-6				X
Kepone ²	143-50-0				X
Famphur ²	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate ³	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene ⁴	70-30-4				X
Diphenylamine ⁵	122-39-4				X

² May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits. Kepone and famphur are not stable in the calibration standard, and searches are made with characteristic ions.

¹ May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits. Normally not reported from Method 8270.

⁴ Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene is searched for in the chromatogram. (See section 12.2.1)

⁵ Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard
(alternate ions may be used)

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane) ¹	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard
(alternate ions may be used)

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate 1 & 2	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263

Table 9

8270B LCS Compounds

LCS Compounds	Spiking Level, ng/ μ L in extract
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/ μ L in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3-Methylphenol ¹	50
4-Methylphenol ¹	50
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

¹3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. For TCLP, results will be reported as 3/4-Methylphenol.

Table 12

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4-Chloro-3-methylphenol	20	50	80	120	160
2-Methylnaphthalene	20	50	80	120	160
Hexachlorocyclopentadiene	20	50	80	120	160
2,4,6-Trichlorophenol	20	50	80	120	160
2,4,5-Trichlorophenol	20	50	80	120	160
2-Chloronaphthalene	20	50	80	120	160
2-Nitroaniline	20	50	80	120	160
Dimethyl phthalate	20	50	80	120	160
Acenaphthylene	20	50	80	120	160
3-Nitroaniline	20	50	80	120	160
Acenaphthene	20	50	80	120	160
2,4-Dinitrophenol	20	50	80	120	160
4-Nitrophenol	20	50	80	120	160
Dibenzofuran	20	50	80	120	160
2,4-Dinitrotoluene	20	50	80	120	160
2,6-Dinitrotoluene	20	50	80	120	160
Diethylphthalate	20	50	80	120	160
4-Chlorophenyl phenyl ether	20	50	80	120	160
Fluorene	20	50	80	120	160
4-Nitroaniline	20	50	80	120	160
4,6-Dinitro-2-methylphenol	20	50	80	120	160
N-Nitrosodiphenylamine	20	50	80	120	160
Azobenzene ²	20	50	80	120	160
4-Bromophenyl phenyl ether	20	50	80	120	160
Hexachlorobenzene	20	50	80	120	160
Pentachlorophenol	20	50	80	120	160
Phenanthrene	20	50	80	120	160
Anthracene	20	50	80	120	160
Carbazole	20	50	80	120	160
Di-n-butyl phthalate	20	50	80	120	160
Fluoranthene	20	50	80	120	160
Benzidine	40	100	160	240	320
Pyrene	20	50	80	120	160
Butyl benzyl phthalate	20	50	80	120	160
3,3'-Dichlorobenzidine	40	100	160	240	320
Benzo(a)anthracene	20	50	80	120	160
Bis(2-ethylhexyl)phthalate	20	50	80	120	160
Chrysene	20	50	80	120	160
Di-n-octylphthalate	20	50	80	120	160
Benzo(b)fluoranthene	20	50	80	120	160
Benzo(k)fluoranthene	20	50	80	120	160
Benzo(a)pyrene	20	50	80	120	160
Indeno(1,2,3-cd)pyrene	20	50	80	120	160
Dibenz(a,h)anthracene	20	50	80	120	160
Benzo(g,h,i)perylene	20	50	80	120	160

Table 13

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-secbutyl-4,6-dinitrophenol (Dinoseb)	20	50	80	120	160
Methyl parathion	20	50	80	120	160
4-Nitroquinoline-1-oxide	20	50	80	120	160
Parathion	20	50	80	120	160
Isodrin	20	50	80	120	160
Methapyrilene	20	50	80	120	160
Aramite 1 and 2	20	50	80	120	160
p-(Dimethylamino)azobenzene	20	50	80	120	160
p-Chlorobenzilate	20	50	80	120	160
3,3'-Dimethylbenzidine	20	50	80	120	160
2-Acetylaminofluorene	20	50	80	120	160
Dibenz (a,j)acridine	20	50	80	120	160
7,12-Dimethylbenz(a)anthracene	20	50	80	120	160
3-Methylcholanthrene	20	50	80	120	160

Table 14

Initial demonstration recovery and precision limits

Compound	Spiking concentration $\mu\text{g/L}$	Limit for Relative Standard Deviation	Limit for average recovery, %
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260 ¹	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

¹ Since the organochlorine pesticides and PCBs are normally determined by method 8080 at Quanterra, they will not be included in the initial demonstration of capability for method 8270B.

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HEALTH AND SAFETY PLAN

HEALTH AND SAFETY PLAN

Introduction

This Health and Safety Plan (Plan) applies to personnel who will potentially be exposed to ground water affected by creosote or coal tar constituents during the retrieval of ground water samples from active pumping wells, the GAC plant, monitor wells, and piezometers. This Plan has been designated to comply with, as a minimum, the requirements set forth in 29 CFR 1910.120, the OSHA standards governing hazardous waste operations. In no case may work be performed in a manner that conflicts with the intent of or the safety concerns expressed in this Plan.

Materials of Concern and Effects of Overexposure

The materials of concern which have been identified for this project are coal tar and creosote-related materials including naphthalene, other polynuclear aromatic hydrocarbons (PAH) and phenolic compounds.

Coal tar and creosote are typically irritating to the eyes, skin and respiratory tract. Acute skin contact may cause burning and itching while prolonged contact and poor hygiene practices may produce dermatitis. Prolonged skin contact with creosote must be avoided to prevent the possibility of skin absorption.

Naphthalene is a hemolytic agent which, upon overexposure to the vapor or ingestion of the solid, may produce a variety of symptoms associated with the breakdown of red blood cells. Naphthalene is also irritating to the eyes and repeated or prolonged contact has been associated with the production of cataracts.

Repeated exposure to certain PAH compounds has been associated with the production of cancer. Contact of PAH compounds with the skin may cause photosensitization of the skin producing skin burns after subsequent exposure to ultraviolet radiation.

Phenolics are generally strong irritants which can have a corrosive effect on the skin and can also rapidly penetrate the skin. Overexposure to phenols and phenolic compounds may cause convulsions as well as liver and kidney damage.

Hazard Assessment

Initial

Because of the relatively low vapor pressures associated with PAH compounds (generally less than 10^{-4} mm Hg at 20°C), they are not expected to present a vapor hazard. The most likely threat of exposure to these compounds will be via skin contact.

Action Limits

The American Conference of Governmental Industrial Hygienists (ACGIH) has established threshold limit values (TLV) for phenol and naphthalene at 5 and 10 ppm, respectively, as 8-hour time weighted averages (TWA). Based on these values, the action limits in Table 1 have been set. The lower limit of 5 ppm is based on the TLV for phenol while the upper limit of 50 ppm is based on a minimum protection factor of 10 for a half-mask, air purifying respirator.

TABLE 1

Action Limits for Air Contaminants

Limit	Persistent Concentration in the Breathing Zone	Procedure
Lower	5 ppm	Don respirators, step up monitoring
Upper	50 ppm	Stop work and back off from immediate work area until levels subside in the breathing zone

Response

When the PID yields persistent breathing-zone readings at or above the lower action limit, workers in the affected area will don respirators. Air sampling will continue on a more frequent basis. If readings are persistent at or above the upper limit, workers shall back off from the immediate work area until measured breathing-zone concentrations fall below the lower limit, at which time operations will resume and normal air monitoring will continue. If breathing zone levels do not fall below the upper limit, workers are to leave the work area and report the condition immediately to the City, the Engineer, or its representative. If necessary, engineering controls will be instituted to maintain vapor concentrations below the upper limit or arrangements will be made to upgrade to Level B protection.

Personal Protective Equipment

Personal protective equipment (PPE) will be donned, as necessary, based on the hazards encountered. Listed below is the PPE to be utilized during this project and the conditions requiring its use.

PPE

- Coveralls - Polyethylene coated Tyvek if work involves contact with affected soil or groundwater
- Boots - Chemical resistant type if work involves contact with affected soil or groundwater
- Hard hat - When working in the vicinity of operating heavy machinery
- Face shield - If splash hazard exists

- Gloves - Nitrile for potential contact with affected soil or groundwater
- Respirator - MSA Comfo II with GMC-H Cartridges if PID reading exceeds 5 ppm or if dust or odors become objectionable
- Chemical safety goggles - If eye irritation occurs

Because of the carcinogenicity of certain PAH compounds, and because of the skin hazards associated with PAH and phenolic compounds, it is important that appropriate protective clothing be worn during work activities, which may involve the possibility of skin contact with affected soil or groundwater. As a minimum, the presence of visible creosote or coal tar-related material shall constitute evidence of affected soil or groundwater.

Health and Safety Training

Personnel covered by this Plan must have received appropriate health and safety training prior to their working on the site. Training will include:

- Requirements for and use of respirators and PPE
- Required personal hygiene practices
- Requirements for employees to work in pairs
- Proper material handling
- Proper sampling procedures
- Maintenance of safety equipment
- Effective response to any emergency
- Emergency procedures
- Hazard zones
- Decontamination methods
- General safety precautions

A copy of the Standard Safety Procedures (Table 2) will be given to each worker covered by this Plan.

TABLE 2

Standard Safety Procedures

Employees are required to work in pairs
Wash face and hands prior to eating, smoking, or leaving the site
No smoking or eating is allowed in the work area during excavation or sampling activities
Wearing of contact lenses is not permitted in the work area
Contaminated material (e.g., Tyvek coveralls) must be properly disposed of before leaving the site
All work must be conducted in accordance with local, state and federal EPA and OSHA regulations, particularly 29 CFR 1910.120

Decontamination

Administrative procedures require hygienic practices consistent with work hazards. employees will be instructed in the training program on proper personal hygiene procedures.

Contaminated, reusable PPE, such as boots, hard hats, face shields and goggles, will be decontaminated prior to leaving the site. The decontamination procedure follows:

- Rinse with water to remove gross contamination
- Wash in Alconox or equivalent detergent solution
- Rinse with clean water

Contaminated, disposable PPE, such as Tyvek coveralls and gloves will be placed in 55-gallon drums and stored while arrangements are made for disposal.

Respirators, if used, will be cleaned and disinfected after each day of use. The face-piece (with cartridge removed) will be washed in a hypochlorite (or equivalent) disinfecting solution, rinsed in warm water and air dried in a clean place.

Emergency Procedures

This Plan has been established to allow site operations to be conducted without adverse impacts on worker health and safety as well as public health and safety. In addition, supplementary emergency response procedures have been developed to cover extraordinary conditions at the site.

General

All accidents and unusual events will be dealt with in a manner to minimize a continued health risk to site workers. In the event that an accident or other unusual event occurs, the following procedure will be followed:

- First aid or other appropriate initial action will be administered by those closest to the accident/event. This assistance will be conducted so that those rendering assistance are not placed in a situation of unacceptable risk. In the event that a worker is caught in a trench collapse, call for emergency assistance immediately.
- All accidents/unusual events must be immediately reported to the Owner.
- All workers on site should conduct themselves in a mature, calm manner in the event of an accident/unusual event, to avoid spreading the danger to themselves, surrounding workers and the community.

Responses to Specific Situations

Emergency procedures for specific situations are given in the following paragraphs.

Worker Injury

If an employee in an affected area is physically injured, Red Cross first-aid procedures will be followed. Depending on the severity of the injury, emergency medical response may be sought.

If the injury to the worker is chemical in nature (e.g., overexposure), the following first-aid procedures are to be instituted:

- **Eye Exposure** - If affected solids or liquids get into the eyes, wash eyes immediately using large amounts of water and lifting the lower and upper lid occasionally. Obtain medical attention immediately.
- **Skin Exposure** - If affected solids or liquids get on the skin, promptly wash the affected skin using soap or mild detergent and water. Obtain medical attention immediately when exposed to concentrated solids or liquids.
- **Inhalation** - If a person inhales large amounts of a toxic vapor, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Obtain medical attention as soon as possible.
- **Swallowing** - When affected solids or liquids have been swallowed, the Poison Control Center will be contacted and their recommended procedures followed.

Emergency Notification

In an extraordinary event that might be damaging to personnel or adjacent property, immediate notification of the proper emergency service will be required. The proper emergency service is determined by the nature of the emergency.

EMERGENCY NOTIFICATION

Fire Department	911
Ambulance	911
Police Department	911
Methodist Hospital	932-5000
Poison Control Center	347-3141

OTHER CONTACTS

MPCA - Miriam Horneff	612-296-7715
EPA - Darryl Owens	312-886-7089
City of St. Louis Park - Scott Anderson	612-924-2557
- William Gregg	612-924-0117

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COMMUNITY RELATIONS PLAN

COMMUNITY RELATIONS PLAN

The Sampling Plan is to be completed in accordance with the Consent Decree-Remedial Action Plan for Reilly Tar & Chemical Corporation's St. Louis Park, Minnesota, N.P. L. Site. All community relations programs related to this work will be coordinated through the following agencies:

United States	Ms. Denise Gawlinski United States Environmental Protection Agency (312) 886-9859
State of Minnesota	Ms. Katherine Carlson Minnesota Pollution Control Agency (612) 297-1607
City of St. Louis Park	Ms. Lynn Schwartz City of St. Louis Park (612) 924-2521

Information necessary to conduct the Community Relations Plan will be provided by the City and Reilly Industries, Inc.